

University Of Alberta



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LABORATORY MANUAL

for
Chemistry:
Experiments
and
Principles

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MacNab
Haenisch
McClellan
O'Connor

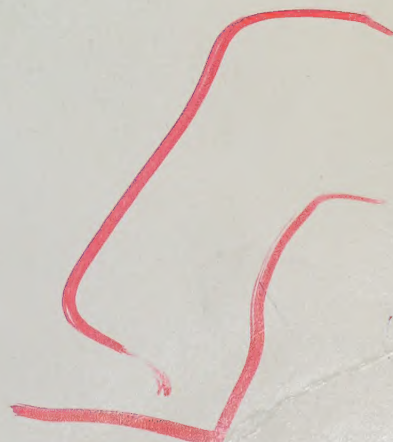
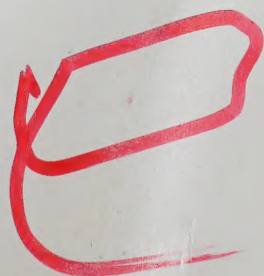
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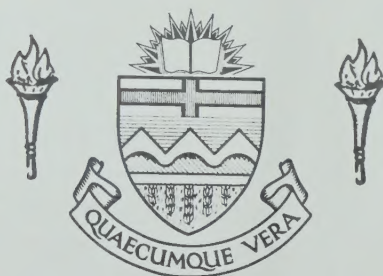
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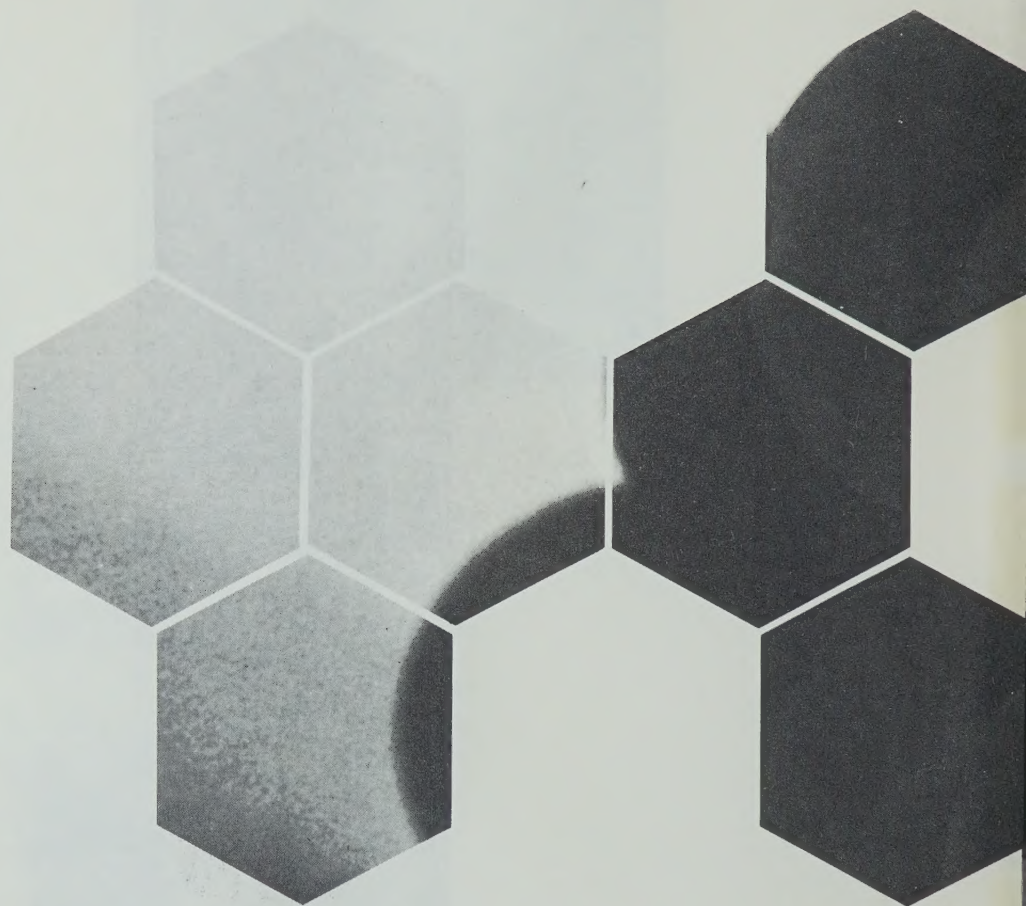


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and Principles

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Preface

To The Student

The experiments in this lab manual are closely meshed with the text, CHEMISTRY — EXPERIMENTS AND PRINCIPLES. Many of the experiments are used to provide data and experimental background before the topics are discussed in the text. As you learn to recognize and to use some of the important principles and concepts of chemistry, you will be in a better position to appreciate and understand the theories which are proposed to explain the regularities revealed by the experiments.

These experiments:

- 1) will help you make your own discoveries of the regularities and principles which unify the study of chemistry and make it easier to understand;
- 2) emphasize the making of careful observations and quantitative measurements under controlled experimental conditions;
- 3) stress the preparation of well-organized data tables and the processing of data so that you can readily make deductions and recognize the regularities that exist;
- 4) contain challenging, thought-provoking questions which will help you apply the principles you have learned to new situations.

In addition to the important background information obtained from the experiments, the important processes of scientific investigation and many laboratory techniques will be experienced. Familiarize yourself with each assigned experiment before coming to the laboratory, and prepare data tables in your notebook so that you will not have to refer to your manual while you are in the laboratory. Read the directions on page x for writing laboratory reports and those on page ix for laboratory instructions and safety precautions.

You will gain self-reliance by making each experiment a job well done. Do your part when working with a partner. Work together as a team, each contributing something to the investigation. Use ingenuity and common sense. You will find there are many opportunities for logical and imaginative thinking. Many experiments contain suggestions for additional investigations which may be undertaken with the permission of your teacher.

The Appendix contains an illustrated section on manipulating glass tubing which may be helpful to you. Other sections discuss briefly the metric system, uncertainty in measurement, and some mathematics which will be needed to make simple calculations. Several useful tables are also available.

Remember that your laboratory work is the core of your chemistry course. You have a unique and challenging opportunity to observe firsthand many of the regularities of chemistry. Out of such experimentally obtained observations,

scientists have developed the principles that unify chemistry and the theories which provide the current explanations. This is scientific method in action.

To The Teacher

This new laboratory manual for CHEMISTRY — EXPERIMENTS AND PRINCIPLES is one of the authorized revisions of the CHEM Study laboratory manual for CHEMISTRY — AN EXPERIMENTAL SCIENCE. Many of the CHEM Study experiments have been retained, some have been modified, some deleted, and some new experiments have been added. We retain the same approach to the study of chemistry that was developed in the former work: chemistry is based on experiments.

Much more than our own teaching experience is contained herein. Many suggestions from teachers who have taught the CHEM Study course have been used. We gratefully acknowledge the many contributions that have been made by our colleagues and friends who were involved in the production of the original CHEM Study materials.

We would like to take this opportunity to express our appreciation to the late Professor Lloyd E. Malm. His leadership in developing the CHEM Study lab manual has had a great influence on high school chemistry. His friendship, advice, and inspiration are gifts that have benefited us greatly.

J. E. Davis, Jr.
W. K. MacNab
E. L. Haenisch
A. L. McClellan
P. R. O'Connor

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Laboratory Instructions and Safety Precautions

1. The laboratory is a place for serious work. Horseplay can never be tolerated.
2. Always prepare for an experiment by reading the directions in the manual before you come to the lab. Follow the directions implicitly and intelligently, noting carefully all precautions. Do not alter an experiment until after receiving the approval of your teacher.
3. **Do only the experiments assigned or approved by your teacher. Unauthorized experiments are prohibited.**
4. If an acid or other chemical is spilled, wash it off immediately with cool water.
5. Do not touch chemicals with your hands unless directed to do so. Wash your hands before leaving the lab.
6. Never taste a chemical or solution.
7. When observing the odor of a substance, do not hold your face directly over the container. Fan a small amount of the vapor toward you by sweeping your hand over the top of the container.
8. Allow ample time for hot glass to cool. Remember hot glass looks like cool glass. Bathe skin burns in cool water.
9. Smother small fires with a towel. Be sure you know the location of the fire extinguisher in the lab.
10. **Report any accident, even a minor injury, to your teacher.**
11. Wear protective eye goggles and protective aprons at all times in the lab. Use the fume hood when directed.
12. Throw all solids and paper to be discarded into a waste jar or wastebasket. Never discard matches, filter paper, or any slightly soluble solids, in the sink.
13. Check the label on a reagent bottle carefully before removing any of its contents. Read the label twice to be sure you have the right bottle.
14. Never return unused chemicals to the stock bottles. Do not put any object into a reagent bottle except the dropper with which it may be equipped.
15. Keep your apparatus and desk area clean.

Laboratory Reports

Research chemists keep a notebook as a record of their experiments. You should do the same. A permanent record of your lab observations will help you answer many questions.

Your lab reports will often concern topics you have not yet discussed in class. This might make them rather difficult to complete. However, many high school students have successfully done this, and you can too. What we are trying to do is to wean you away from the activity involving mere regurgitation of observations or statements. In place of this tedious activity, we are offering you an opportunity to draw your own conclusions from facts you yourself have gathered. Proficiency in creating generalities from raw data is an ability that will serve you well in any walk of life.

Here are a few suggestions that other students have found useful.

1. Record all data as soon as possible after making the observations. Make no erasures; instead, cross out any errors with a single line. Always record your name, the title of the experiment, and the date when the observations were made.
2. Enter all data and observations neatly. Use tabular form whenever it is appropriate. When possible, prepare a data table before coming to the lab.
3. For questions requiring a numerical answer, indicate operations, units, and answer. Carry out arithmetical manipulations at the bottom of the page, or off to the right side. Normally calculations should not be made during the laboratory period.
4. Answer the numbered questions wherever they appear as part of your laboratory report. Use concise statements.
5. Do not include written answers to questions which appear in the introduction and procedure section. Some are intended to direct your attention to problems which will be investigated. Others point out the reason for certain procedures or controls which will enable you to proceed with greater understanding.



Experiment 1

Scientific Observation and Description

Everyone thinks of himself as a good observer. Yet there is much more to observation than meets the eye! It takes concentration, alertness to detail, ingenuity, and often just plain patience. It even takes practice! Try it yourself. See how complete a description you can write about a familiar object — say, a burning candle. Be “scientific” about this and start with an experiment. Observe a burning candle. Should any conditions be controlled? Be ready for surprises here! Sometimes the important conditions are difficult to discover. However, an experiment can be meaningless unless the *conditions that matter* are controlled. Here are some conditions that may be important in *some* experiments but are not important in this one.

The experiment is done on the second floor.

The experiment is done in the daytime.

The room lights are on.

Here are some conditions that *might* be important in your experiment.

The lab bench is near the door.

The windows are open.

You are standing close enough to the candle to breathe on it, but not close enough to singe your hair.

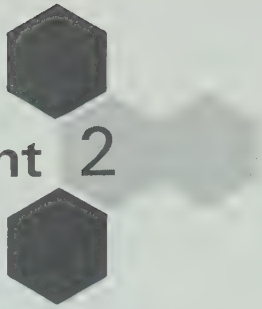
Why are these conditions important? Because they influence the object we wish to observe. Do they have something in common? Yes, there is the common factor that a candle does not operate well in a draft. Important conditions are often not as easily recognized as these. A good experimentalist pays much attention to the discovery and control of conditions that are important.

PROCEDURE

Light a candle and record as many observations as you can during a short period of time (10–15 minutes).

Turn in a copy of your observations to your teacher before you leave the laboratory. Tonight,

rewrite your observations on the burning candle. Consider the points brought out in class discussion and include any further observations you are able to make.



Experiment 2

Observing Evidence of Interaction

Your study of a burning candle in Experiment 1 helped you distinguish between an observation and an interpretation. The difference between a qualitative and a quantitative observation was also considered. After this experience you might agree that there is really much more to making careful observations than was previously realized. Skill and experience *are* needed to be a good observer. The ability to make careful observations is so important that it will be to your advantage to develop this skill as much as possible.

This experiment provides another opportunity for careful observation. Try to think of *conditions that matter*. Be alert to questions that come to mind as you observe. Later in the course there will be opportunity to seek answers to many of the “why” questions that you may begin to wonder about.

Make your observations as complete as you can. Remember to use quantitative as well as qualitative observations. You may wish to use a thermometer to make some quantitative measurements of temperature.

PROCEDURE

Add about two level teaspoonfuls of blue crystals to a 100 ml beaker half full of water. Allow the system of water and crystals to stand a few minutes. Record observations. Then stir the water until the crystals dissolve. After the crystals

dissolve, place a piece of loosely rolled aluminum foil into the liquid.

After listing your observations, write down any questions that occurred to you during this experiment.



Experiment 3

Searching for a Regularity

A regularity is simply something that occurs over and over again. Stones fall when dropped. Ice melts if sufficient energy is added to it. These regularities are based on qualitative observations. They don't tell us how fast stones fall or at what temperature ice melts. Regularities based on quantitative observations would give us this information. In this experiment you will make quantitative observations on the mass and volume of pieces of metal.

The mass of each piece of metal will be determined by weighing. Let us explain the use of the word mass rather than weight. The word *weight* should be used when discussing how much attraction an object experiences when it is in the earth's gravitational field. This attraction is commonly called the force of gravity. The strength of this gravitational field varies slightly from place to place. The weight of an object then depends not only on how much matter it contains, but also on where it is weighed. An object weighs less at the equator than it does at the North Pole. If an object weighs 0.997 lb at the equator, it will weigh 1.002 lb at the North Pole. The weight of an object is determined using a spring balance.

The word *mass*, on the other hand, is used to refer to a property of matter that depends on the number and kind of atoms present. Mass does not vary as the strength of a gravitational field varies. The mass of an object depends on how much matter it contains. Astronauts in space become weightless, but never massless. The mass of an object is determined by matching the object against known masses on a balance. The "known masses" are usually pieces of metal that have been matched against a very carefully preserved set of standards.

Your teacher will give you directions for determining mass appropriate for the balance you will use.

To express amount of mass, we use units called grams. A gram is a unit of mass in the metric system. One pound is equal to a mass of about 454 grams. The mass of a one ounce package of Lifesavers is about 28 grams.

The volume of a solid that is fairly regular in form can be determined by measuring its linear dimensions and manipulating them in an appropriate manner. The volume of a rectangular solid is found by multiplying its length times width times

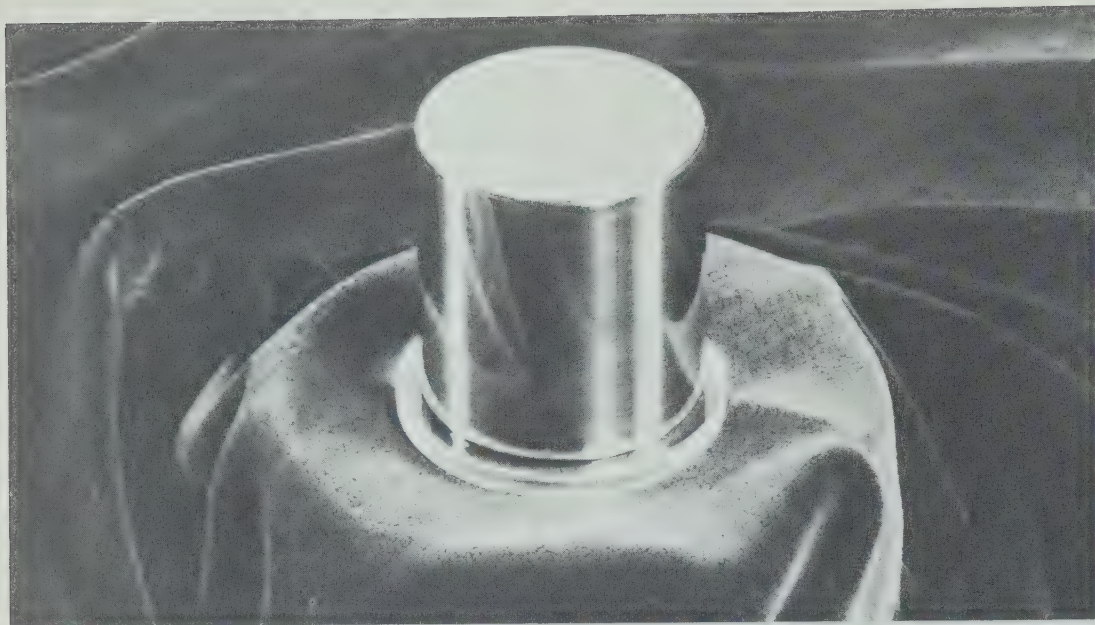


Figure 3-1

The U. S. Prototype Kilogram. It is kept by the Bureau of Standards in Washington, D. C. The International Prototype Kilogram is kept at Sèvres, France. (From U. S. Department of Commerce)

height. However, measuring the linear dimensions of a solid of irregular shape, for example a roughly chipped stone, could be very difficult as well as time consuming. These difficulties make it worthwhile to develop a simple method for determining the volume of solids of irregular shape. One simple technique is to measure the volume of some liquid in which the solid will not dissolve. Then put the solid in the liquid and note the combined volume. The increase in volume is the volume of the solid. A graduated cylinder is a convenient vessel for these measurements.

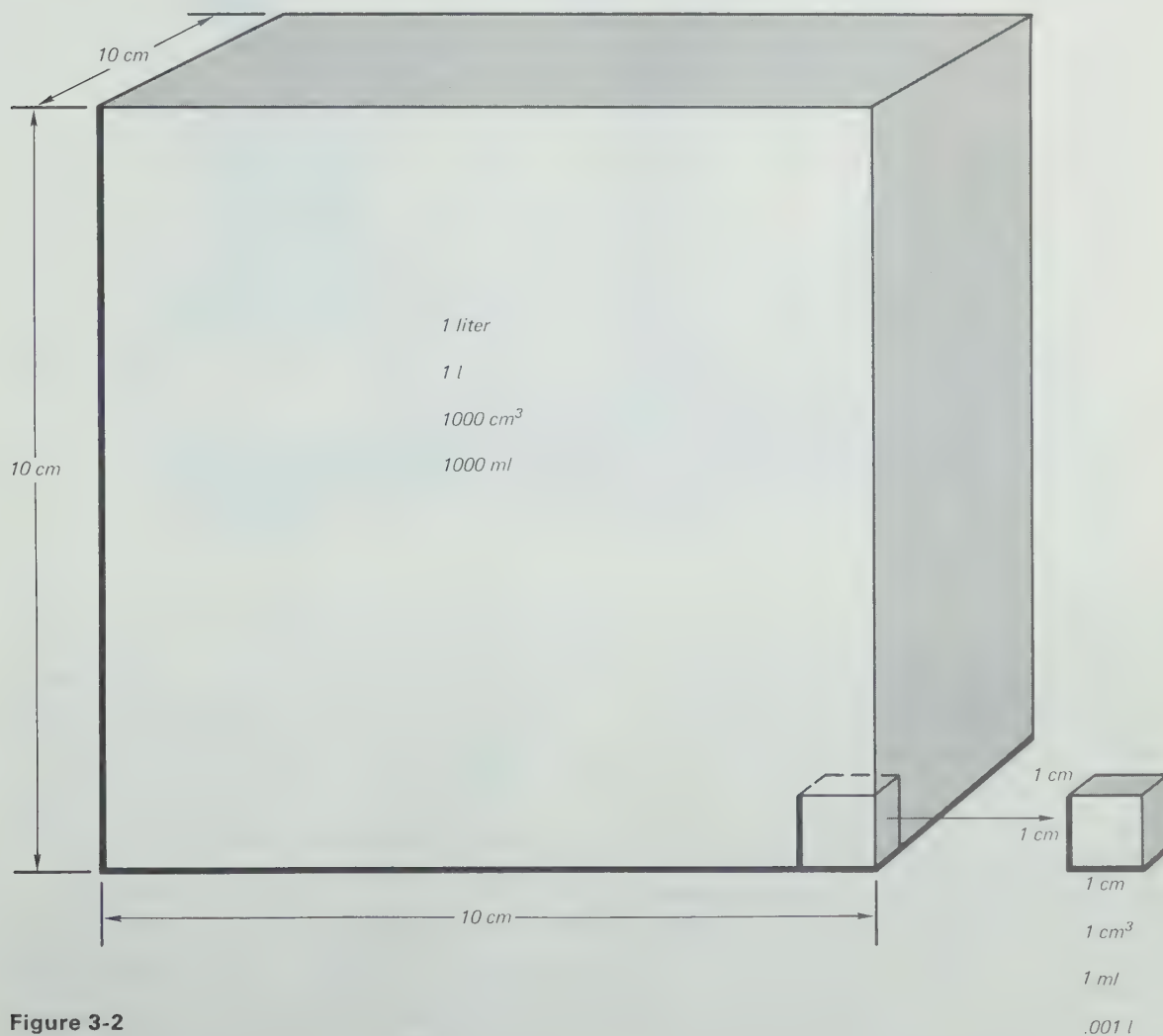
Volume will be expressed in units called either cubic centimeters (cc, cm^3) or milliliters (ml). A liter contains 1000 cm^3 so a ml is the same volume as a cm^3 .

PROCEDURE

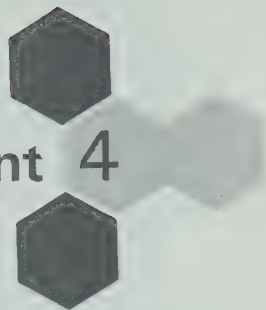
- a. Determine the mass (± 0.1 gram) of a piece of metal.
- b. Determine the volume (± 0.5 milliliter) of the same piece of metal.
- c. Repeat these steps on a sample of another metal. Use the same balance for all weighings in this experiment.
- d. Record the number of your pieces of metal to distinguish them from those of your classmates.

PROCESSING THE DATA

- 1) Obtain mass and volume data from other members of your class. Be careful to keep the pairs of data together.
- 2) Plot these data on a graph. Record mass along the labeled vertical axis and volume along the labeled horizontal axis. Give the graph a title.
- 3) State the regularity, and propose an explanation for what you observed on your graph.
- 4) Should each line go through the point (0, 0) ?
- 5) The introduction to this experiment contains two methods for determining the volume of solids. Which method would give the more precise answer if used on a regular solid? Explain.

**Figure 3-2**

A liter, and a milliliter.



Experiment 4

Weighing an Object Immersed in Two Different Fluids

In this experiment you will investigate the relationship between the mass of an object, its volume, and the environment in which the weighing is done. The mass will then be related to the volume of the object.

Two different objects will be weighed separately, first when immersed in air, and second when immersed in water.

PROCEDURE

- a. Record the number of your objects to distinguish them from those of your classmates.
- b. Weigh each of your two objects in air to the nearest 0.1 gram.
- c. Weigh each of your two objects to the nearest 0.1 g when they are completely immersed in water. Be sure there is no direct contact between the object being weighed and the vessel containing the water. See Figure 4-1.
- d. Make the measurements needed to determine the volume of each object to the nearest 0.1 ml. Use the water displacement method.

PROCESSING THE DATA

- 1) For each object, determine the difference between the apparent mass in air and the apparent mass in water. The reason for using the phrase "apparent mass" rather than "mass" will be clear when this experiment is completed and discussed in class.
- 2) Determine the volume of each object.
- 3) Does the mass of an object or its volume have a greater effect on the difference between the apparent mass of an object in air and its apparent mass in water? Explain.
- 4) Determine the mass of water displaced by each object. One gram of water occupies 1.0 milliliter.

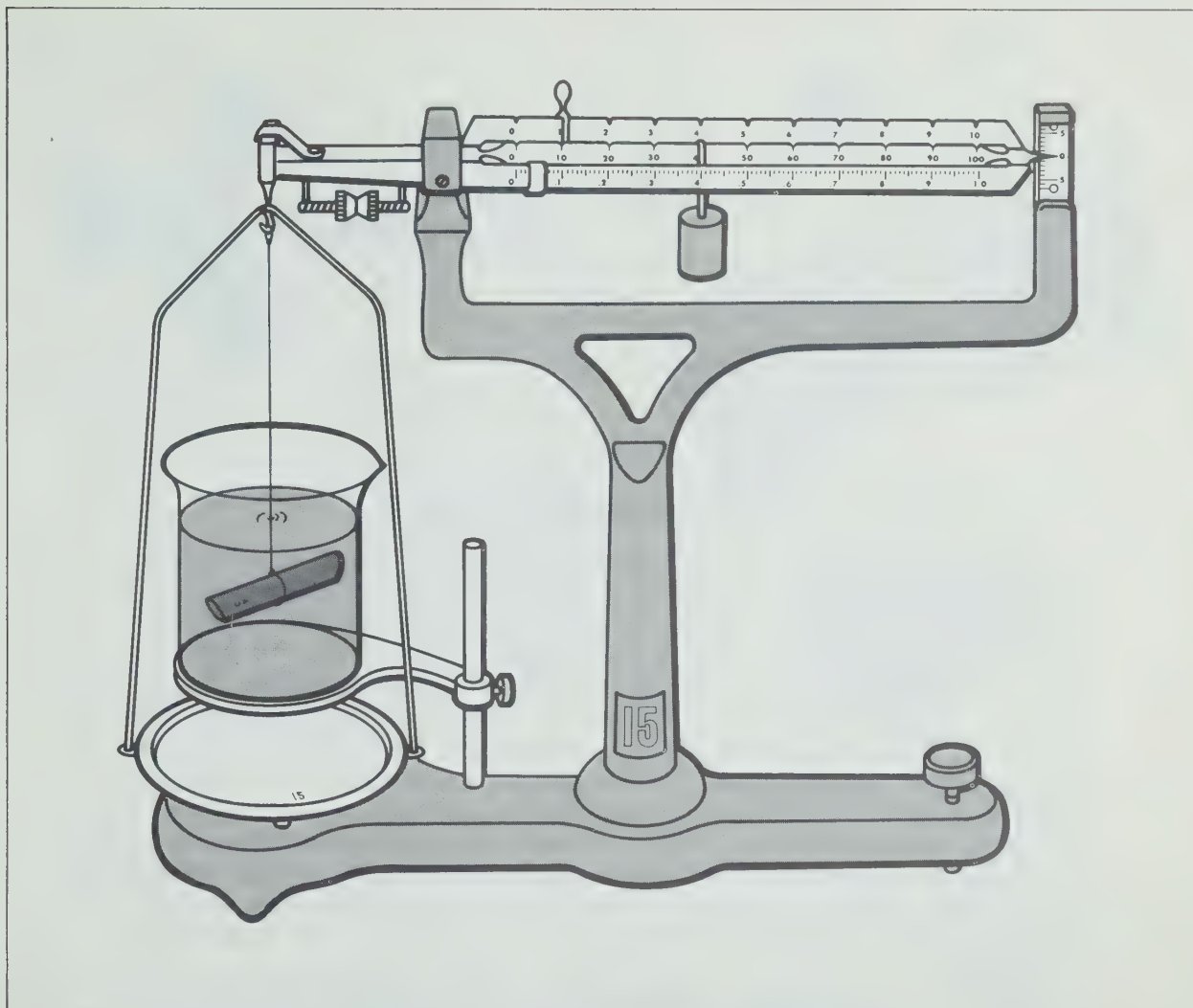
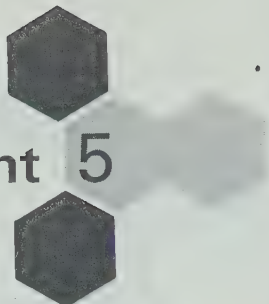


Figure 4-1

Weighing an object immersed in water.

- 5) Determine for each object, the value of the ratio

$$\frac{\text{mass of water displaced}}{(\text{apparent mass in air}) - (\text{apparent mass in water})}$$
- 6) Express in words the significance of the value found for the ratio in item 5 above.
- 7) Will an object have a greater mass in a vacuum or in air? Why?
- 8) One liter of air at room temperature and normal atmospheric pressure has a mass of 1.18 g. What would have been the mass of your objects had they been weighed in a vacuum?
- 9) Why is the adjustment suggested in item 8 above not usually made on weighings made in air?



Experiment 5

The Masses of Equal Volumes of Gases

Everyone knows that solids and liquids have weight. We verify this each time we lift one. We also frequently lift gases, and promptly think nothing of it. When referring to a flask that is full of air, we normally say it is empty. Yet an "empty" 250 ml flask contains about 7×10^{21} molecules. Empty indeed? If the air is withdrawn from the flask, will it weigh less? Your teacher can easily demonstrate this to you. Remember if the weighings are made on a balance in which the flask and contents are matched against known masses, then mass and not weight will have been determined. In this experiment you will determine the mass of equal volumes of several different gases including oxygen and carbon dioxide.

Let us consider some of the conditions that matter for this experiment. You might expect the mass of any volume of gas you can measure on your balance will be rather small. It is. Weighings must be made very carefully. Keep the gas container (plastic bag) clean and dry. A small amount of water can cause a large percent error.

You know the volume a gas occupies depends on its pressure. One gram of gas could fill a one liter flask, or at a reduced pressure, a ten liter flask. If your measurements are to be compared to those of your classmates, pressure will have to be known. A simple way to do this is to open your container of gas to the atmosphere until no gas flow is detected. Then your sample of gas will have the same pressure as the atmosphere, and this can be read from a barometer. Atmospheric pressure usually changes rather slowly. Hence, the pressure will be the same for all volume measurements made during one class period.

Perhaps you also know temperature affects the volume of a gas. As temperature is increased, a gas expands, its volume becomes greater. The temperature of your gas sample must be known. This is easily done by allowing the sample to reach room temperature. Room temperature is usually constant during one class period. Hence, the temperature will be the same for all volume measurements made during one class period.

Before coming to the laboratory read the directions carefully. Prepare a table in your notebook where you can record your data and calculated results.

PROCEDURE**Part I. Weighing Oxygen Gas**

- Obtain a rubber stopper and medicine dropper assembly. Gather the open end of a plastic bag around the large end of the rubber stopper, by folding the bag in small pleats. Hold the bag in place with a rubber band placed over the groove in the stopper. See Figure 5-1.
- Press out any air by smoothing the bag flat. Replace the rubber cap on the medicine dropper. Weigh this assembly to the nearest 0.01 gram.
- Remove the rubber cap and connect the assembly to an oxygen gas source supplied by your teacher. Allow the bag to be fully inflated. See Figure 5-2. Hold the bag assembly by the stopper and disconnect the rubber tubing from the medicine dropper. Allow any excess gas to escape so that the gas in the bag will be at room pressure, but do not squeeze the bag. Then replace the rubber cap.
- Weigh the bag assembly containing the oxygen gas to the nearest 0.01 gram. Take great care that no part of the bag rubs against any stationary part of the balance.

Part II. Weighing Carbon Dioxide Gas

Be sure the bag and stopper assembly is empty and dry. Fill it with carbon dioxide gas using the source supplied by your teacher. Repeat steps c and d of Part I. Try to have the same volume of gas at the same temperature and pressure as before.

Part III. Optional

Obtain the mass of a bag full of another gas.

Obtaining the Volume of the Bag

- Empty the bag and fill it with air by blowing into it. Try to have the same volume of gas as you used in each of the previous experiments.

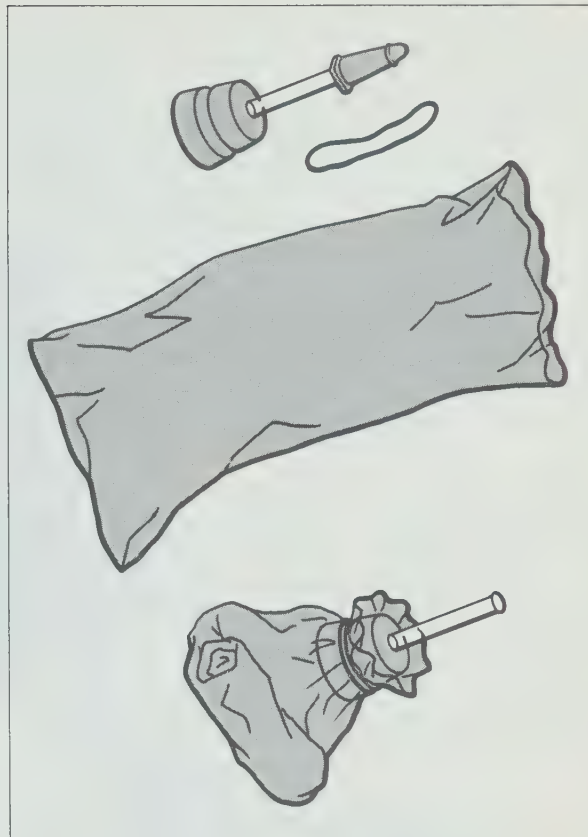


Figure 5-1

The plastic bag assembly.

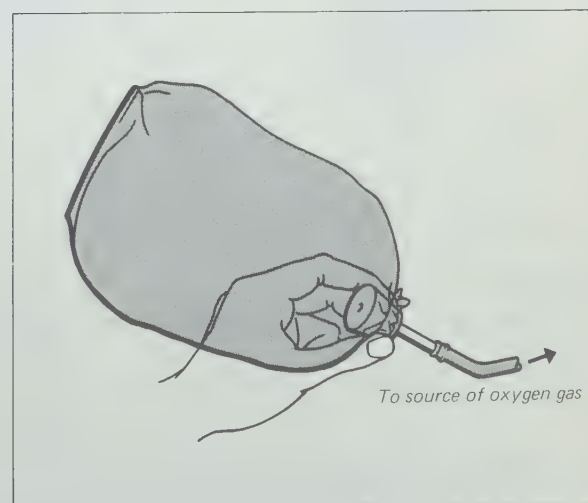


Figure 5-2

Filling the plastic bag with oxygen gas.

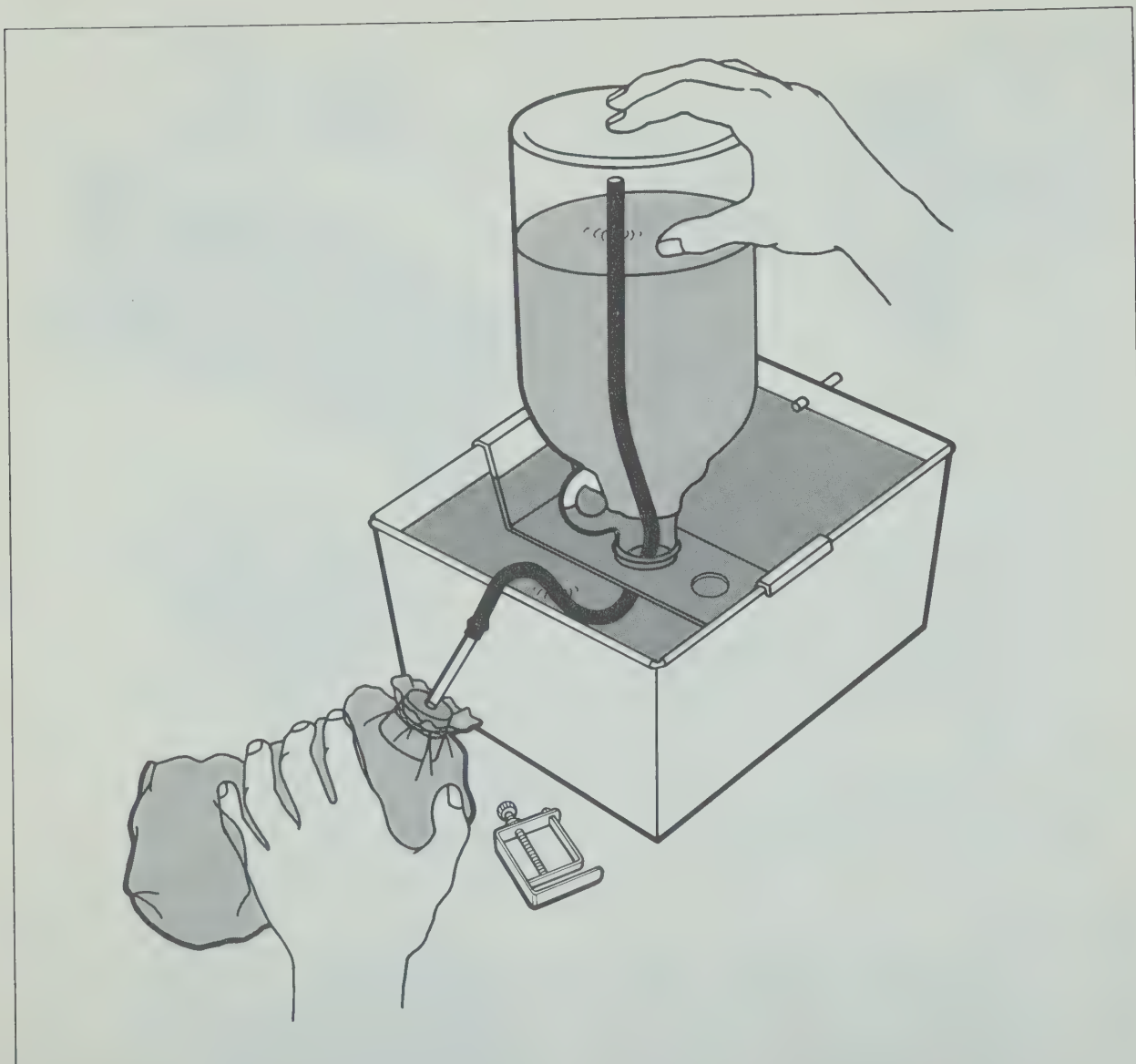


Figure 5-3

Transferring gas from bag to bottle. One volume of water is displaced from the bottle for each volume of gas that enters the bottle.

- f. Determine the volume of air contained in the bag by the method shown in Figure 5-3. Completely fill a large bottle or jug (about $\frac{1}{2}$ gallon size) with tap water. Fit it with a stopper and invert it in a large container of

water. Remove the stopper under the water. Obtain a rubber hose and a pinch clamp. Fasten the clamp near one end of the hose and push the other end up into the bottle.

- g. Remove the cap from the medicine dropper. In its place attach the free end of the rubber tubing from the bottle of water. Remove the clamp and gently press on the bag to force the gas into the bottle. Finally flatten out the bag to remove all the gas.
- h. Pinch the tubing closed and remove it from the water container. Place a stopper in the mouth of the bottle. Remove the bottle from the water and set it upright on the table.
- i. Measure the amount of water required to refill the bottle, using the largest graduated cylinder available to you. Record the volume of the water needed.
- j. Record the room temperature and pressure.

The Effect of Buoyancy of Air on Weighings

Look back to your notes for Experiment 4. In that experiment you found an object weighed less when submerged in water than it weighed in air. The difference we say is due to the buoyancy of

the water, and was equal to the mass of the water displaced. A similar situation exists when we weigh objects submerged in air. They are buoyed up by a force equal to the mass of the air displaced. This force is usually so small it is ignored. A one liter object weighed in air is buoyed up by a force of about 1.2 grams. One liter of air has a mass of about 1.2 grams. The object weighs 1.2 grams more in a vacuum than in air. The object actually contains 1.2 more grams of matter than the weighing in air reveals. The object has an apparent mass which is 1.2 grams less than its actual mass.

Since a liter of most solids weighs at least 1000 grams, the small error of 1.2 grams is often ignored. One liter of a gas however weighs so little that 1.2 grams cannot be ignored. The amount your sample of gas was buoyed up must be considered. The buoyant force depends on the mass of a liter of air, and this varies with temperature and pressure. Table 5-1 provides the needed data.

TABLE 5-1

Mass of 1 Liter of Air, at the Conditions Shown

Pressure (mm Hg)	15°C	20°C	25°C	30°C	35°C
600	0.97 gram	0.95 gram	0.94 gram	0.92 gram	0.90 gram
610	0.98	0.97	0.95	0.93	0.92
620	1.00	0.98	0.97	0.95	0.93
630	1.02	1.00	0.98	0.97	0.95
640	1.03	1.01	1.00	0.98	0.97
650	1.05	1.03	1.01	1.00	0.98
660	1.06	1.05	1.03	1.01	1.00
670	1.08	1.06	1.04	1.03	1.01
680	1.10	1.08	1.06	1.04	1.03
690	1.11	1.09	1.08	1.06	1.04
700	1.13	1.11	1.09	1.07	1.06
710	1.14	1.12	1.10	1.09	1.07
720	1.16	1.14	1.12	1.10	1.09
730	1.18	1.16	1.14	1.12	1.10
740	1.19	1.17	1.15	1.13	1.12
750	1.21	1.19	1.17	1.15	1.13
760	1.23	1.20	1.18	1.16	1.15
770	1.24	1.22	1.20	1.18	1.16

PROCESSING THE DATA

- 1) What is the apparent mass of oxygen in the bag? (Subtract the mass of the empty bag from the mass of the bag full of oxygen.)
- 2) Calculate the mass of the air displaced by the bag filled with gas.
- 3) What is the actual mass of oxygen in the bag? (Add the mass of the air displaced to the apparent mass recorded from the balance.)
- 4) In a similar way determine the actual mass of carbon dioxide gas in the bag and the actual mass of any other gas used.
- 5) Compare the actual mass of each gas measured to that of oxygen by dividing each mass by the mass of the same volume of oxygen. Express each ratio as a decimal fraction.

A QUESTION TO WONDER ABOUT

Is there any relation between the masses of equal volumes of gases and the relative mass of molecules?

ADDITIONAL INVESTIGATIONS

To be undertaken as extracurricular experiments. Consult your teacher before proceeding.

- 1) How do the masses of equal volumes of gases at atmospheric pressure change as the temperature is increased?
- 2) The burner gas in the laboratory is usually natural gas, which is mostly methane, CH_4 , but contains some ethane, C_2H_6 , and small amounts of other compounds. Weigh a sample of burner gas and calculate the percent of methane and of ethane, assuming that they are the only gases present.



Experiment 6

Iron-Copper Sulfate Reaction

In this experiment you will let iron filings react with a copper sulfate solution. An excess of copper sulfate will be used so that all of the iron will be reacted. By weighing the amount of iron used and the amount of new substance formed you will be able to determine a quantitative relation between reactants and products for this reaction.

As you do the experiment, record your data carefully and neatly. Take special care to show the units as an important part of each measurement. Express each measurement using the correct number of significant figures as determined by the precision of the measuring instrument.

Before coming to the laboratory, you should plan what you are to do. A suitable data table is a big help. Use the entries suggested at the end of the procedure section. This advance preparation will save you time and allow you to concentrate on making the required observations without wasting time.

PROCEDURE

- a. Weigh an empty 100 ml beaker to the nearest 0.01 gram.
- b. Set the balance to read 10 grams heavier than the beaker. Add enough copper sulfate crystals, as shown in Figure 6-1, to make the pan just go down. You need not determine this mass precisely.
- c. Add about 25 ml of distilled water to the copper sulfate crystals.
- d. Place the beaker on a ring stand and warm the solution until it just starts to boil. Be careful not to let the solution boil over. See Figure 6-2. Remove the burner.

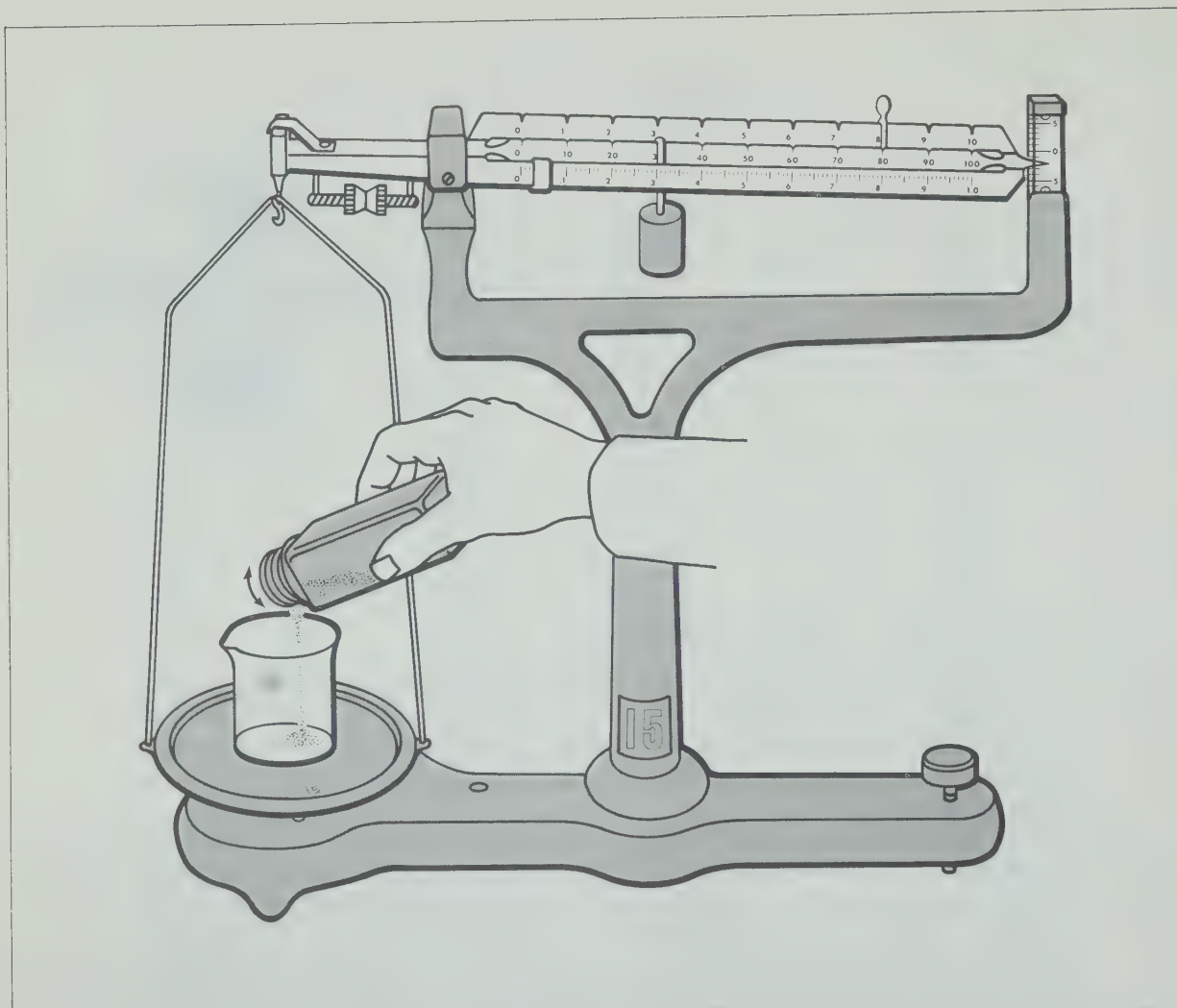


Figure 6-1

Weighing approximately 10 grams of hydrated copper sulfate crystals.

- e. Weigh a piece of weighing paper as shown in Figure 6-3A. Record the mass in your data table. Set the balance to indicate 2 grams heavier than the weighing paper. Add enough iron filings to make the pan just go down. See Figure 6-3B. Determine the exact mass to the nearest 0.01 gram. Record the mass as iron filings plus weighing paper in your data table.
- f. While stirring the hot copper sulfate solution with a glass rod, add the iron filings a little at a time.
- g. Carefully decant the solution. *Decant* means to pour off the liquid, along a stirring rod, leaving solid behind as shown in Figure 6-4. Wash the solid product several times with distilled water. Use about 10 ml of water for

- each washing. Carefully decant after each washing.
- h. After the final washing, the solid must be dried. Your teacher will suggest a suitable method. Be sure to label your beaker uniquely.
- i. After the solid is dry, allow the beaker to cool to room temperature. Weigh the beaker and dry solid. Be sure to use the same balance for all weighings. To make sure the sample is dry, you should subject it to further drying and weigh it again.

Your Data Table Should Include the Following:

mass of empty beaker
mass of weighing paper
mass of iron filings plus weighing paper
mass of product (dry solid) plus beaker

Figure 6-3A

Determining the mass of a piece of weighing paper.

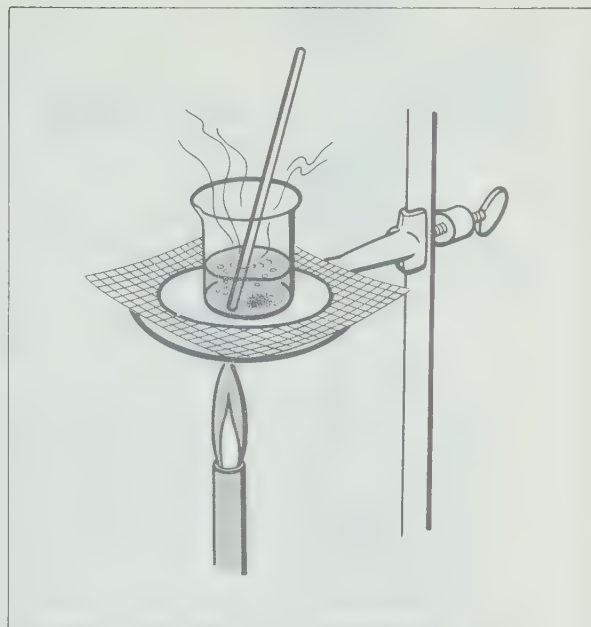
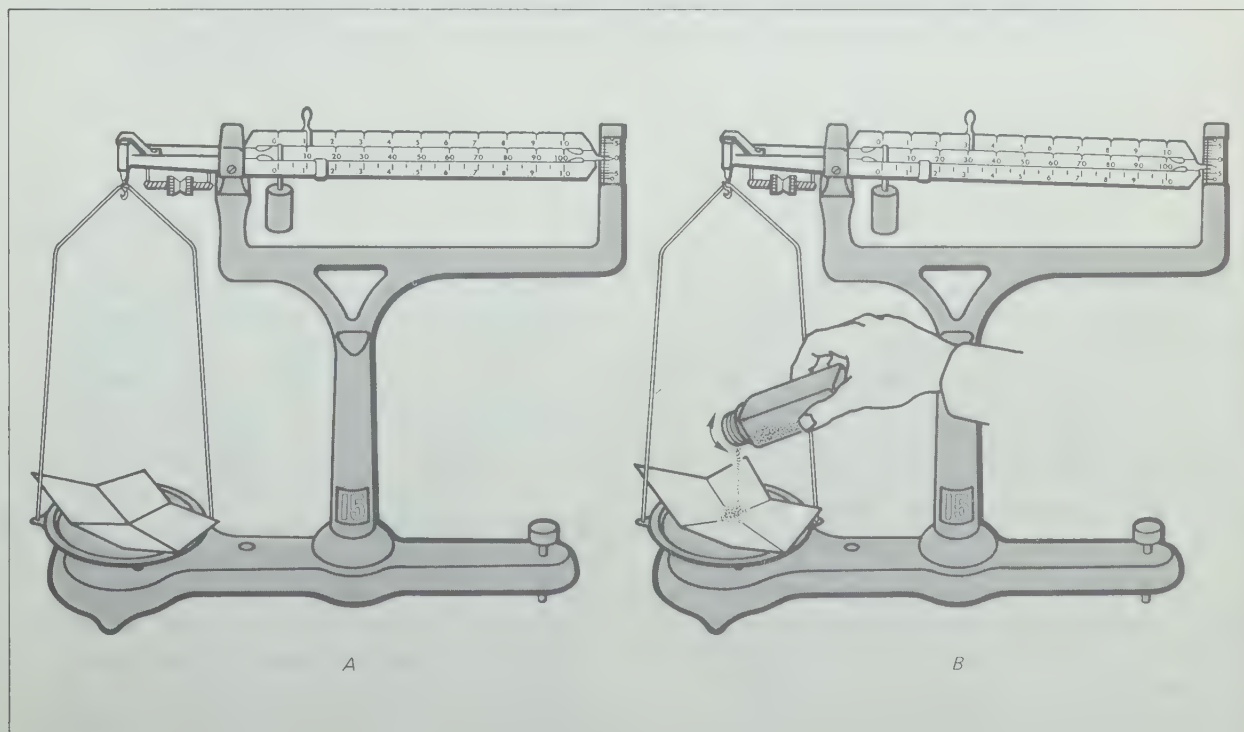


Figure 6-2 Warming the copper sulfate solution.

Figure 6-3B

Weighing about 2 grams of iron filings to nearest 0.01 gram.

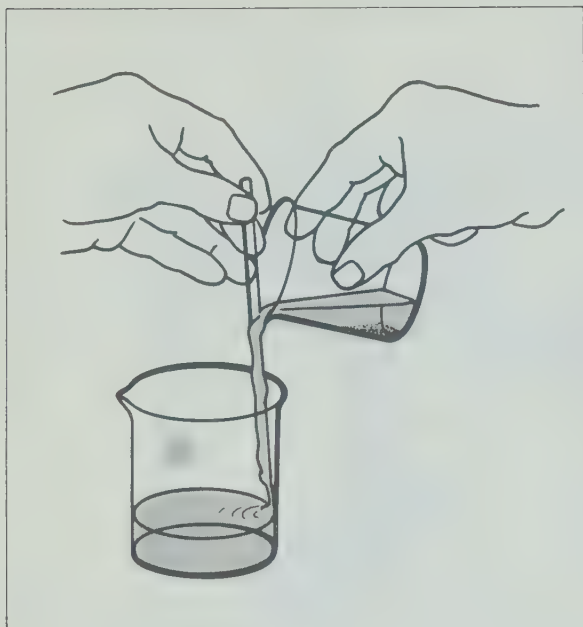


Figure 6-4

Decanting a liquid from a solid.

PROCESSING THE DATA

- 1) Determine the mass of the product.
- 2) Determine the moles of iron used. Recall that the mass of a substance divided by the mass per mole equals the number of moles.
- 3) Assuming the product to be copper, determine the moles of copper produced.
- 4) Divide the moles of iron, Fe, by the moles of copper, Cu, to determine the mole ratio, Fe/Cu. Be sure to express your answer as a decimal and use the correct number of significant figures.
- 5) What evidence did you observe to indicate that some of the copper in solution remained unchanged?



Experiment 7

Copper-Silver Nitrate Reaction

In this experiment you will weigh a sample of solid silver nitrate and prepare a water solution of it. You will also weigh a piece of coiled copper wire, place it in the silver nitrate solution, and observe its behavior. By weighing the copper wire at the close of the experiment you will be able to determine the amount of copper reacted. Using these and other measurements you will be able to determine a quantitative relation between reactants and products.

In keeping with chemical practice, we will refer to the chemical substances by using appropriate symbols. Copper is an *element*; it contains only one kind of atom. The symbol for copper is Cu. Silver nitrate is a *compound* and has the formula AgNO_3 . The symbol Ag is for the silver part and the symbols NO_3 for the nitrate part. The group of atoms NO_3 , consisting of one nitrogen atom and three oxygen atoms, is often found in chemical compounds and has the name *nitrate*.

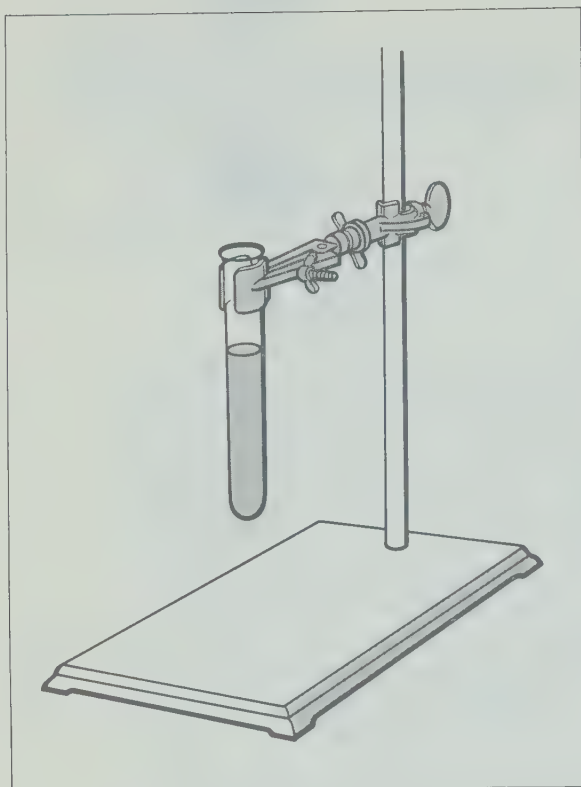
Before coming to the laboratory, prepare a table in your notebook so that you can record the data you will observe. Use the entries suggested at the end of the procedure section.

PROCEDURE

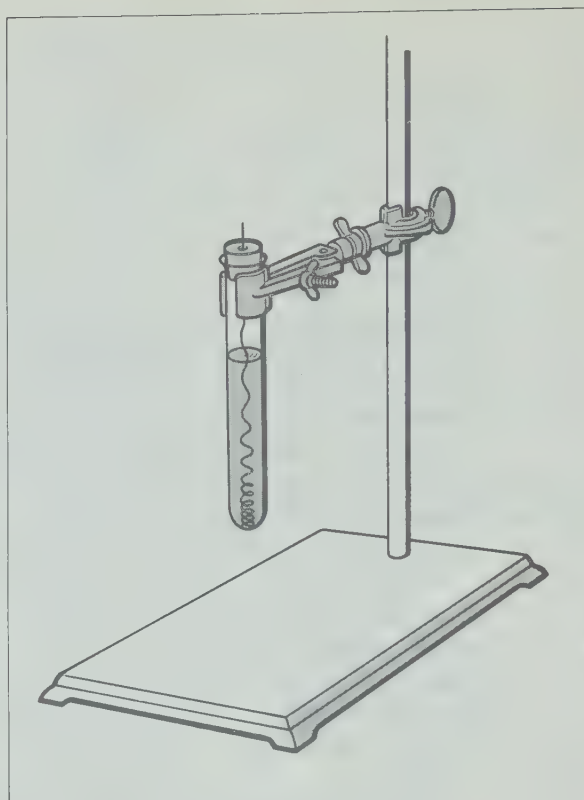
- Obtain a 30 cm length of bare copper wire, a clean dry 100 ml beaker, an 18×150 mm test tube, and a vial containing about 3 grams of silver nitrate crystals.
- Weigh the clean dry beaker and the vial containing silver nitrate. Determine all masses to the nearest 0.01 gram.
- Measure about 25 ml of distilled water into the test tube. Add the solid silver nitrate from the vial to the water in the test tube. Do not rinse or wash the vial. Save it to be weighed as directed in step f. (**Caution!** Be careful not to get any of the silver nitrate, solution or solid, on your skin or clothing.

It will interact with them.) Fasten the tube in a clamp on a ring stand as shown in Figure 7-1. Stir the solution with a stirring rod to dissolve the crystals and mix the solution. Drain the stirring rod by touching the end of the rod to the inside of the test tube near the mouth. Rinse the stirring rod with a small amount of distilled water into the test tube.

- Coil the copper wire by wrapping it around a glass stirring rod. See Figure 7-2A. Stretch the coiled wire as shown in Figure 7-2B until it is about 2 cm longer than the test tube. Be sure to leave the wire tightly

**Figure 7-1**

Test tube containing silver nitrate solution clamped in place.

**Figure 7-3**

Coiled copper wire placed in silver nitrate solution.

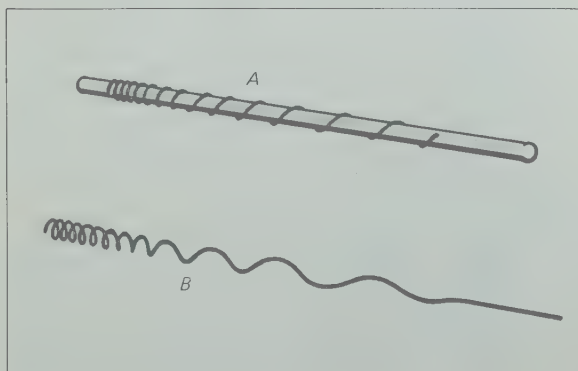
coiled near one end and stretched out and
straightened toward the other end.

Figure 7-2A

Coiled copper wire.

Figure 7-2B

Coiled copper wire, stretched.



- Place the coiled wire into the silver nitrate solution with the closely coiled end at the bottom of the test tube. Place a one hole rubber stopper in the test tube with the end of the wire extending through the hole. See Figure 7-3. Be careful not to shake or in any way disturb the contents of the test tube. Record observations in your laboratory notebook. Allow the reaction to continue for 30 minutes.
- While the reaction is proceeding, weigh the empty vial with cap in order to determine the mass of silver nitrate used in the experiment.
- Shake the crystals from the copper wire. Allow the wire to remain in the solution for a few seconds to see if the reaction is complete. Using a wash bottle, rinse the wire

into the weighed 100 ml beaker. See Figure 7-4. Empty the contents of the test tube into the 100 ml beaker. Add a few ml of water to transfer all the crystals from the test tube. Dip the copper wire in acetone, and then set it aside to dry. Weigh the copper wire when it is dry.

- h. Let the crystals of silver settle in the beaker and carefully decant the solution. *Decant* means to pour off the liquid, leaving solid behind. Pour the liquid along a glass stirring rod as shown in Figure 7-5. Wash the residue three or four times using about 10 ml of distilled water each time. Carefully decant after each washing. A few very tiny particles of silver may float off as you decant. They have a very small mass and may be neglected.
- i. After the final washing, the sample must be dried. Your teacher will suggest a suitable method. Be sure to identify your beaker uniquely.
- j. After the residue is dry, allow the beaker to cool to room temperature. Weigh the beaker containing the product. Make all weighings

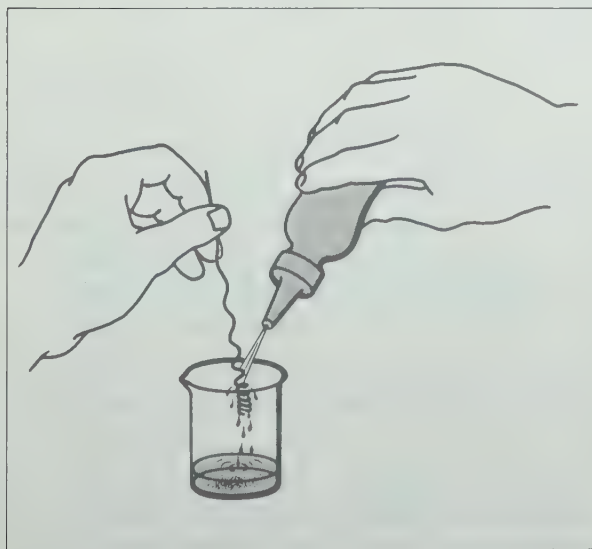


Figure 7-4

Rinsing the copper wire using a wash bottle.

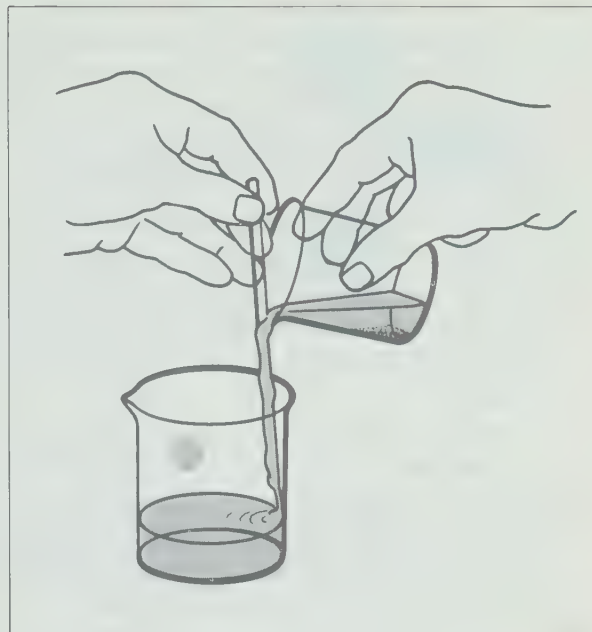


Figure 7-5

Decanting the liquid from a solid.

on the same balance. To make sure the sample is dry, you should subject it to further drying and weigh it again.

- k. Cover the beaker with a watch glass and save the dry silver crystals. They will be used later in Experiment 8.

Your Data Table Should Include the Following:

mass of clean dry 100 ml beaker
mass of vial, cap, and silver nitrate
mass of empty vial with cap
mass of the coiled copper wire before reaction
mass of the coiled copper wire after reaction
mass of the beaker plus silver crystals

PROCESSING THE DATA

- 1) Determine the change in mass of the copper wire during the experiment.

- 2) Determine the number of moles of copper which reacted. Recall that the mass of a substance divided by the mass per mole equals the number of moles.
- 3) Determine the mass of silver obtained.
- 4) Determine the number of moles of silver.
- 5) Divide the moles of silver by moles of copper to determine the ratio Ag/Cu . Be sure to express your answer as a decimal and to the correct number of significant figures.
- 6) Determine the moles of AgNO_3 used in the experiment.
- 7) Divide the moles of silver by moles of silver nitrate to determine the ratio Ag/AgNO_3 . Express your answer as a decimal using the correct number of significant figures.
- 8) Using the results of your experiment, write whole number coefficients in the following statement:
One mole of Cu plus _____ mole(s) of AgNO_3 in solution produces _____ mole(s) Ag plus one mole of $\text{Cu}(\text{NO}_3)_2$ in solution.
- 9) How many atoms of copper were removed from the wire used in your experiment?
- 10) How many atoms of silver metal were formed in your experiment?

A QUESTION TO WONDER ABOUT

What caused the color in the solution to appear as the reaction proceeded?



Experiment 8

Conservation of Mass During Chemical Change

In this experiment the silver produced in Experiment 7 will be reacted with 6 *M* nitric acid, HNO_3 , to form a water solution of silver nitrate, AgNO_3 . Then by reacting this solution with a solution of sodium chloride, NaCl , you will obtain a silver chloride, AgCl , precipitate.

After the products have been washed and dried, the mass of reactants (silver nitrate and sodium chloride) can be compared to the mass of the products (silver chloride and residue).

Before coming to the laboratory, prepare a table in your laboratory notebook so that you can record the data you will observe. Use the entries suggested at the end of the procedure section.

PROCEDURE

Part I. Preparation of Solid Silver Nitrate

Before starting the experiment, obtain and record the following data from Experiment 7:

- mass of beaker used in Experiment 7
- mass of silver nitrate used in Experiment 7
- mass of silver produced in Experiment 7

- a. Obtain your beaker containing the silver crystals prepared in Experiment 7. Number the beaker #1.
- b. Place beaker #1 in a fume hood. Add about 10 ml of 6 *M* nitric acid, HNO_3 , to the silver. You may have to warm the acid to start the reaction. Avoid inhaling any of the poisonous reddish brown fumes of nitrogen dioxide, NO_2 , which form. Leave the beaker in the

fume hood as directed by your teacher. Your teacher will make arrangements to evaporate the solution to dryness.

- c. Next laboratory period weigh the beaker containing silver nitrate. Use the same balance for all weighings. To make sure the sample is dry, you should subject it to further drying and weigh it again.
- d. Add about 15 ml of distilled water to the crystals weighed in step c.

Part II. Preparation of Solid Silver Chloride

- e. Weigh a clean dry 100 ml beaker and label it #2. Adjust your balance to indicate about 2 grams more than the mass of the beaker. Add enough NaCl to the beaker, as shown

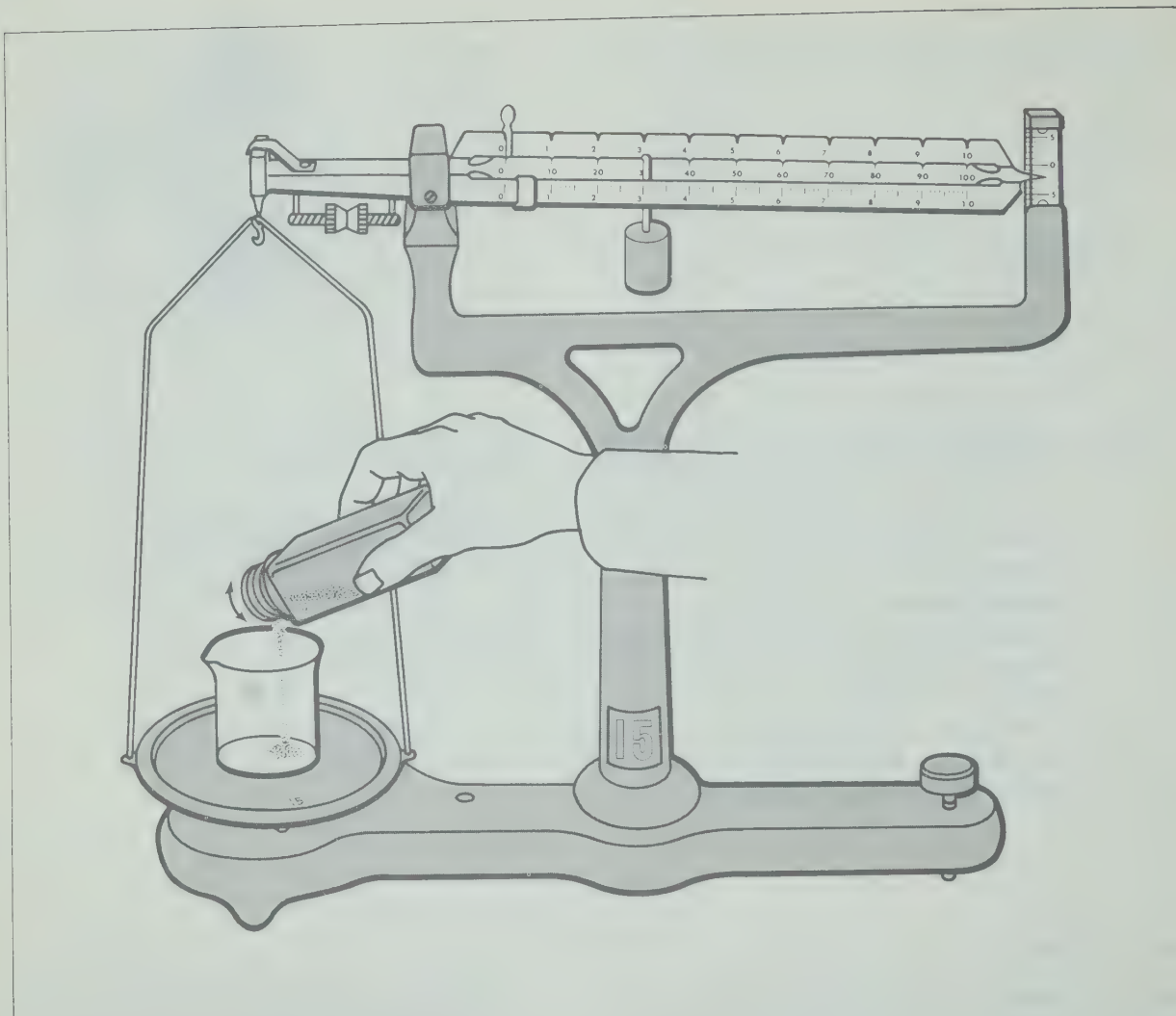


Figure 8-1

Weighing about 2 grams of sodium chloride to the nearest 0.01 gram.

in Figure 8-1, to make the pan just go down. Determine the mass of the NaCl and beaker to the nearest 0.01 gram. Dissolve the salt in beaker #2 with about 15 ml of distilled water. Stir until all of the crystals are dissolved.

- f. Slowly add the sodium chloride solution from beaker #2 to the silver nitrate solution in beaker #1 as shown in Figure 8-2. (Note

this shows another technique for transferring liquid. See Figure 6-4.) Add a few drops. Stir. Add a few more drops. Stir. Add a few milliliters. Stir. Continue until all the solution has been added. The solid formed is called a *precipitate*. It is silver chloride.

- g. Rinse the empty beaker (#2) with about 5 ml of distilled water from a wash bottle. Direct the water around the inside of the

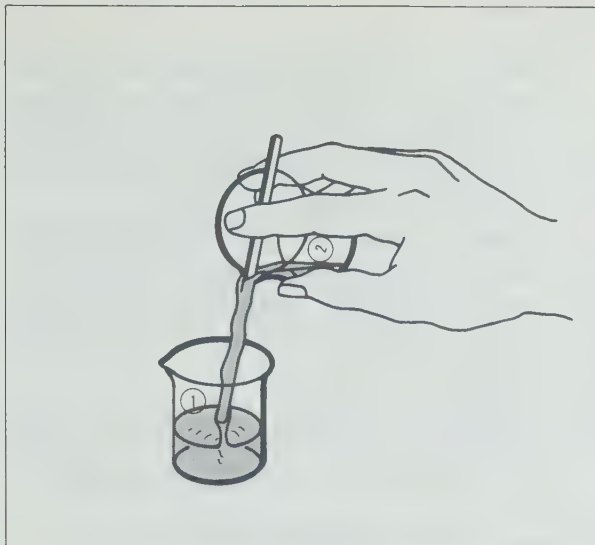


Figure 8-2

Pouring sodium chloride solution into silver nitrate solution.

beaker as shown in Figure 8-3. Add the rinse water to the mixture in beaker #1. Rinse beaker #2 again with another 5 ml of

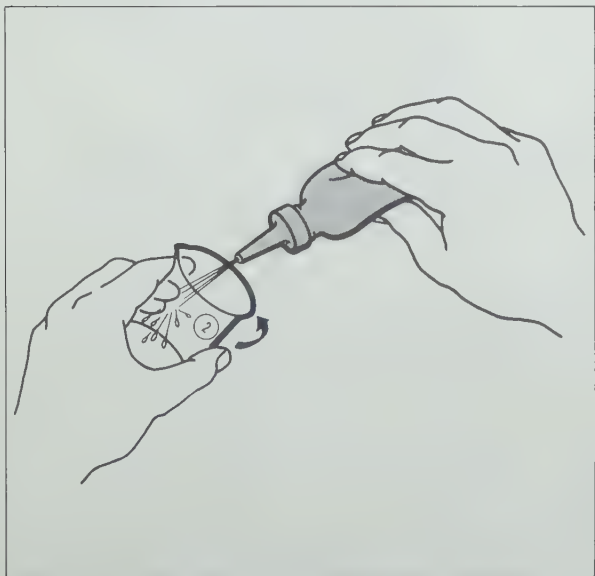


Figure 8-3

Rinsing the beaker using a wash bottle.

- distilled water. Keep beaker #2 to be used later in the filtering step of the procedure.
- h. Heat the resulting precipitate and the solution to boiling or until the solution becomes clear.
- i. Determine the mass of a filter paper to the nearest 0.01 gram. Fold the paper and tear off one corner as shown in Figure 8-4. Fit the paper into a funnel and moisten it with some distilled water from a wash bottle. Gently press the filter paper snugly into the funnel. Set up the funnel for filtering as shown in Figure 8-5. Place the torn off corner in the funnel.
- j. Place beaker #2 under the funnel. The tip of the funnel should touch the beaker so a steady stream of liquid can run down the side. Decant the clear liquid from beaker #1 into the funnel. A small amount of the precipitate may transfer to the filter paper, but try to keep most of it in the beaker where it can be washed more readily. The solution that passes through the filter paper is called the *filtrate*.
- k. Wash the precipitate in the beaker with about 15 ml of distilled water, stirring with a glass rod to aid the washing. Decant the wash water into the funnel. Repeat the washing procedure with another 15 ml of

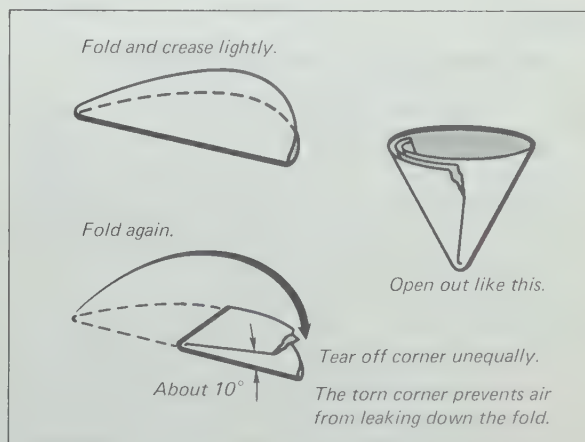


Figure 8-4

Folding and tearing filter paper for snug fit in funnel.

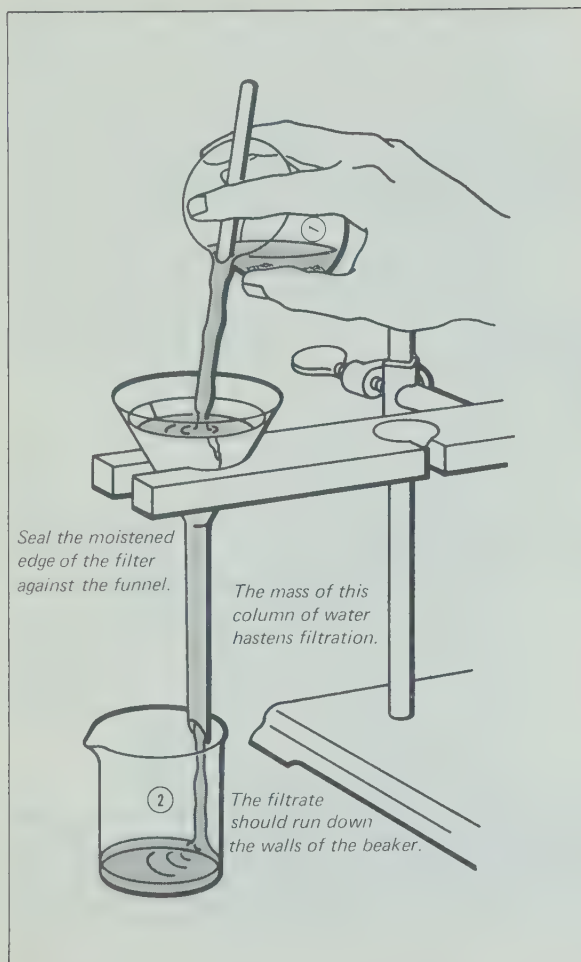


Figure 8-5

Decanting the liquid from beaker #1 into the filter.

water. Decant the wash water again into the funnel. After the washing and filtering is complete, place the filter paper and any solid it contains in beaker #1 which contains most of the precipitate.

- l. Check to make sure each beaker is labeled. Then place both samples, the filtrate in beaker #2 and the wet precipitate and filter paper in beaker #1, in the place designated by your teacher for drying.
- m. Weigh both beakers containing the dry samples and record the masses. Save the silver chloride as directed by your teacher.

Your Data Table Should Include the Following:

mass of beaker #1 used in Experiment 7
 mass of silver nitrate used in Experiment 7
 mass of silver from Experiment 7
 mass of beaker #1 and solid AgNO_3 made in Part I of this experiment
 mass of beaker #1, filter paper, and solid AgCl
 mass of beaker #2
 mass of one filter paper
 mass of beaker #2 and solid residue

	Mass (g)	Number of Moles
Ag from Experiment 7		
AgNO ₃ used in Experiment 7		
AgNO ₃ produced in Experiment 8		
NaCl		
AgCl in beaker #1 (be sure to subtract mass of filter paper)		
residue in beaker #2		

PROCESSING THE DATA

- 1) Reproduce the above table in your notebook and make the necessary calculations to fill in the entries.
- 2) How does the mass of AgNO₃ produced in this experiment compare with the mass used in Experiment 7? How do you account for any similarity or difference?
- 3) Determine the value of the ratio :
$$\frac{\text{mass (AgCl + residue)}}{\text{mass (AgNO}_3 + \text{NaCl)}}$$
Express your answer as a decimal using the correct number of significant figures. Compare your results with those from others in your class. What can you conclude?
- 4) Compare your results for the number of moles of silver used, of silver nitrate produced in Part I, and silver chloride produced in Part II. This comparison can be made by

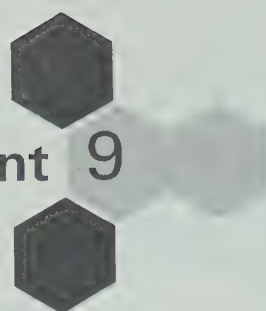
computing the ratio between moles of silver and each of the other substances. Use whole numbers to express your comparison. What can you conclude about the number of moles of silver containing compounds in this series of chemical changes?

- 5) Calculate the percentage of silver reclaimed in this experiment. Start with moles of AgNO₃ used in Experiment 7 and finish with moles of AgCl produced in Experiment 8.

QUESTIONS TO WONDER ABOUT

Pure silver nitrate is a white solid. How do you account for any color which may have been present in your sample?

What causes silver compounds to darken in color when exposed to light?



Experiment 9

The Formula of a Hydrate

Many salts which have been crystallized from a water solution appear to be perfectly dry, yet when heated yield large quantities of water. The crystals change form, even color sometimes, as the water is driven off. This suggests that water was present as part of the crystal structure. Such compounds are called **hydrates**. The number of moles of water present per mole of anhydrous salt is usually some simple number. One example is the hydrate copper sulfate. Its blue crystals look and feel dry. Yet, each mole of hydrate contains five moles of water. Its formula is $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

In this experiment you will be given an appropriate hydrate selected by your teacher. You will determine the mass of water driven off by heating and the amount of anhydrous salt which remains. Your teacher will give you the mass of 1 mole of the anhydrous salt so that you can find the empirical formula of the hydrate.

PROCEDURE

- In your notebook, organize a data sheet to record data and calculations.
- Place a clean dry crucible with cover in a triangle mounted on an iron ring. Leave the cover askew so that any water in the crucible can easily escape. Heat with a Bunsen burner flame for two or three minutes. Remove the flame.
- When the crucible is cool enough to touch, transfer the crucible and cover to a balance.
- Record their mass to the nearest 0.01 gram.
- Put enough of the hydrate crystals in the crucible to fill it one-fourth to one-third full. Weigh the crucible, cover, and contents.
- Place the crucible with its cover only slightly askew, on the triangle and heat them very gently so as to avoid spattering. Continue heating gently until the popping and sizzling have stopped. Gradually increase the flame

until the crucible bottom is at most a dull red. Adjust the cover until there is a 0.5 cm opening between it and the crucible. See Figure 9-1. Maintain this temperature for five minutes. Use a pair of tongs to transfer carefully the hot crucible and cover to a desiccator. See Figure 9-2. A desiccator is a container in which a very dry atmosphere is maintained. Water is usually removed from the air by an anhydrous salt which combines with the water to form a stable hydrate. Your teacher will tell you which anhydrous salt is in your desiccator. See Figure 9-3.

- f. Wait about five minutes, until the crucible is cool enough to touch. Transfer it and the cover to the balance and weigh them.
- g. To make sure all the water is driven off, heat the crucible and its cover to dull redness again. Carefully transfer them to a desiccator. When cool, weigh again. If your results do not agree within 0.03 gram, consult your instructor concerning further heating and weighing.

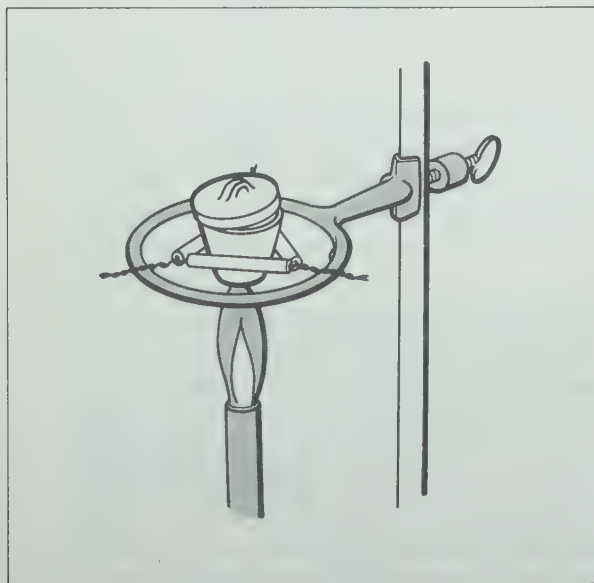


Figure 9-1

Heating a crucible and cover.

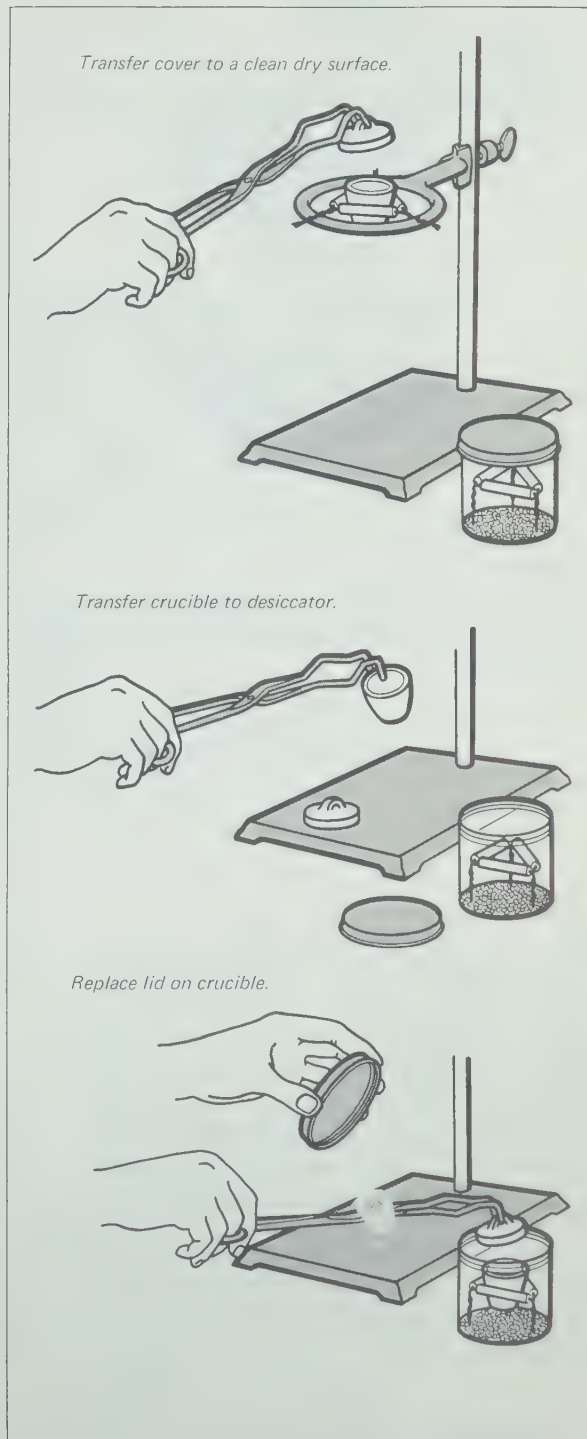


Figure 9-2

Transferring a hot crucible and cover.

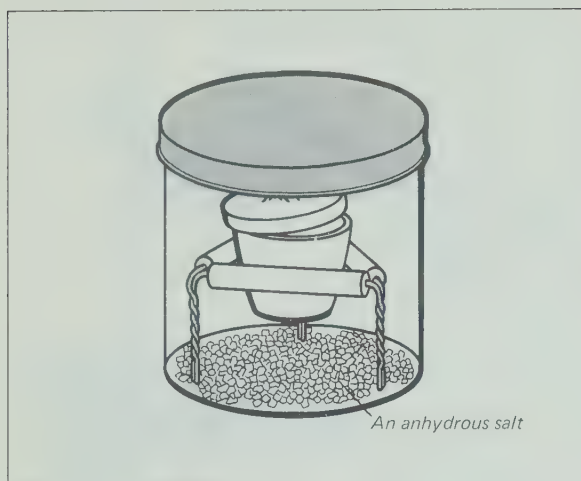


Figure 9-3

Crucible cooling in the dry atmosphere in a desiccator. (The lid must be kept tightly screwed on the desiccator whenever possible.)

Your Data Should Include the Following Information:

number of the balance you use
mass of crucible and cover
mass of crucible, cover, and hydrate
mass of crucible, cover, and anhydrous salt
after first heating
mass of crucible, cover, and anhydrous salt
after second heating
mass of 1 mole anhydrous salt (from teacher)

PROCESSING THE DATA

- 1) Calculate the number of moles of the anhydrous salt you prepared.
- 2) Calculate the number of moles of water driven from your sample of hydrate.
- 3) Calculate the moles of water per one mole of anhydrous salt.
- 4) Write the empirical formula for the hydrate.
- 5) Can you suggest reasons why the above method might not be suitable for all hydrates?



Experiment 10

Temperature-Volume Relation

You have already learned about the pressure-volume relation of a gas and have seen how a study of this important regularity helped develop a particle model of matter. Is there also a simple temperature-volume relation? What will happen to a gas filled rubber balloon if it is warmed or cooled? If a gas is held in a cylinder by a piston which is free to move up or down, what will happen if the cylinder is warmed? What will happen if the cylinder is cooled? How much does the volume change for each Celsius degree temperature change? Does the volume change in a regular way? Does the volume vary directly with change in temperature or perhaps inversely?

This experiment will help you find answers to these questions. The cylinder you will use is a glass capillary tube. The piston is a mercury plug which traps a small volume of air in the tube. See Figure 10-1.

You can determine the volume of the trapped air by attaching the glass tube to the side of a thermometer and using the scale on the thermometer as shown in Figure 10-2. Since volume is proportional to the length of a cylinder, the length of the air column can be used to represent the volume in arbitrary units.

PROCEDURE

- Obtain a thin wall capillary tube containing a mercury plug as shown in Figure 10-1. Hold the tube along a thermometer as shown in Figure 10-2A. Note the lower end of the tube is opposite the lower end of the thermometer.
- Make a mark with a glass marking pen on the capillary tube opposite the zero mark on the thermometer. Move the tube up along the thermometer to measure the length l_1 (in degree units) as shown in Figure 10-2B. Record this length and fasten the tube against the thermometer as shown in Figure 10-2A. Use a piece of tape to fasten the tube near the upper end and a small rubber band near the lower end. The lower end of the tube is opposite the lower end of the thermometer.
- Allow the tube and the thermometer to stand for a few minutes in order to come to room temperature. Record this temperature. Determine the length l_2 (in degree units) from the zero mark on the thermometer to the bottom of the mercury plug. See Figure 10-2A. Hold the thermometer in a vertical position while measuring l_2 .
- Let the tube and the thermometer stand in an ice-slush bath for a few minutes with the tube immersed all the way to the mercury plug. See Figure 10-3. Record the temperature and the length l_2 .
- Repeat step d using a boiling water bath in place of the ice-slush. Be sure the tube is held vertically and immersed to the plug while measuring l_2 .

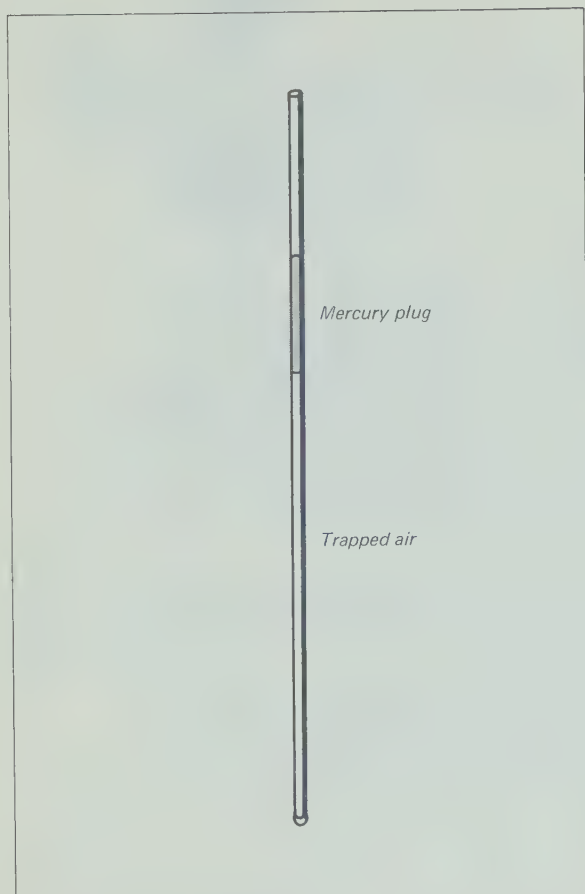


Figure 10-1

Capillary tube containing air and plug of mercury.

PROCESSING THE DATA

- 1) Determine the total length of the air column ($l_1 + l_2$) for each temperature. If we assume the inside diameter of the capillary to be constant, we can use the length of the air column to represent the volume. Since $(l_1 + l_2) \times \text{cross-sectional area} = \text{volume of trapped air}$, we can represent the volume as $(l_1 + l_2)$ in arbitrary units. The arbitrary unit is the volume of a cylinder having the diameter of the mercury plug and the length of one degree on the thermometer scale.

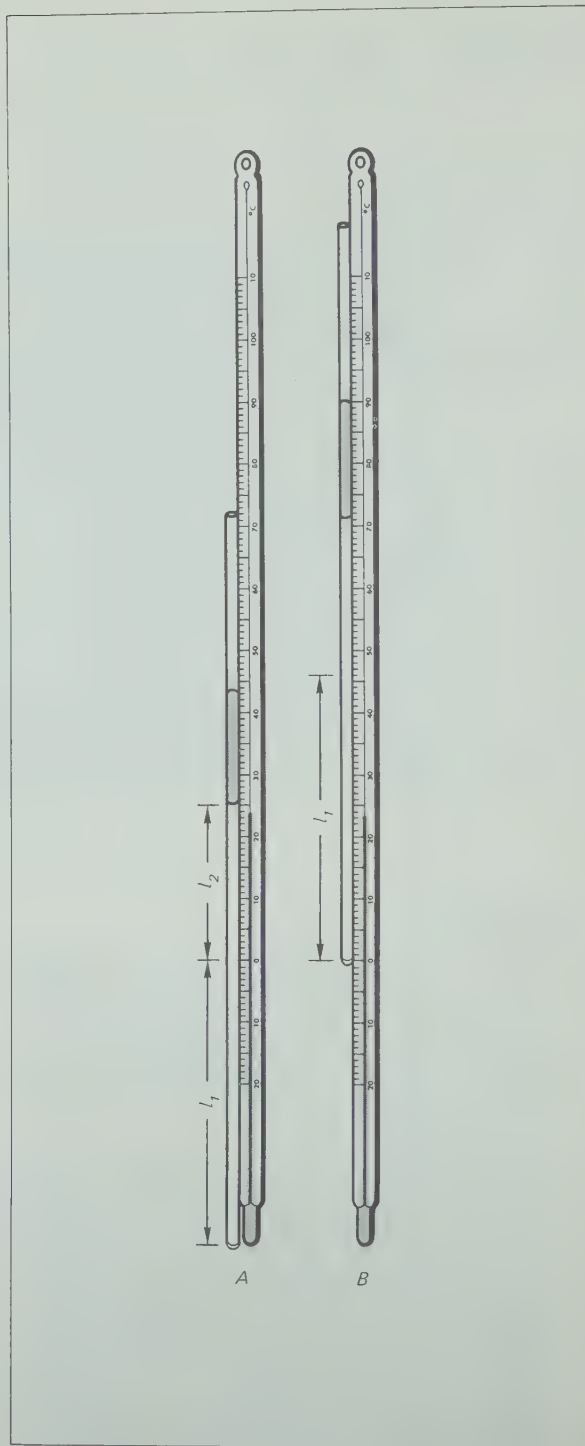


Figure 10-2

Measuring length of air column trapped in the capillary tube.

- 2) Plot the volumes and temperatures on a graph. Present volume along the vertical axis (short side of paper) and temperature along the horizontal axis (long side of paper). Plot the volume scale with zero and the temperature scale with -300°C at the origin. Draw the best straight line through the three points.
- 3) By *extrapolation* (extending the line beyond the plotted data) determine the temperature at which the line intersects the horizontal axis. Give reasons why a gas does not behave as suggested by the extrapolated line.

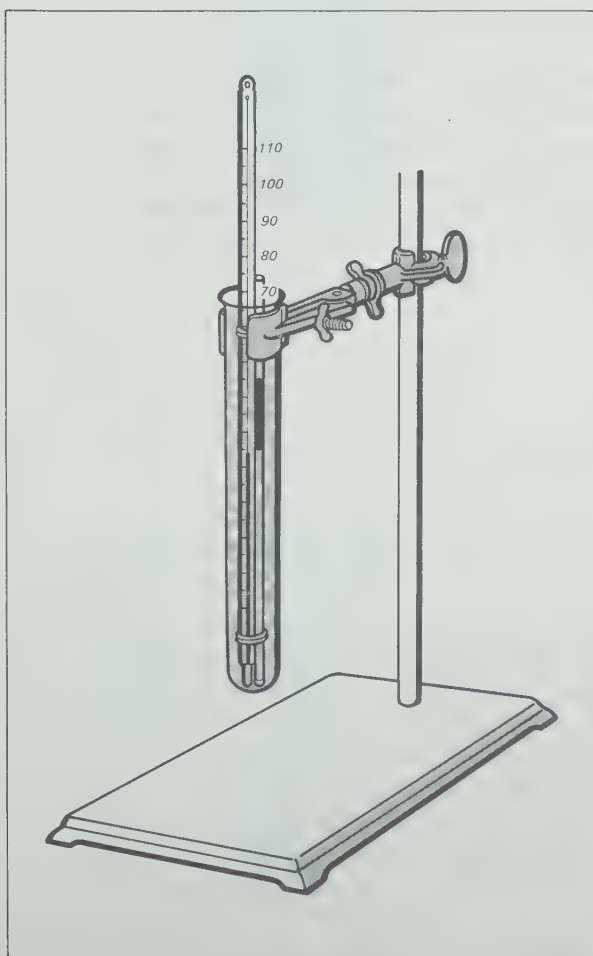


Figure 10-3

Thermometer and capillary tube immersed in ice-slush bath.

- 4) Express the temperature-volume relation as a mathematical expression.

ADDITIONAL ACTIVITY

Determine the pressure-volume relation of a gas.

PROCEDURE

- a. With the capillary tube attached to the thermometer as shown in Figure 10-2A, lay the thermometer flat on the table. Determine the length of the air column with the tube in this horizontal position. Also measure the length of the mercury plug in millimeters.
- b. Hold the thermometer vertically with the *closed end* of the capillary tube downward. Determine the length of the air column with the tube in this position.
- c. Turn the thermometer and the tube upside down with the *open end* of the capillary tube downward. Determine the length of the air column with the tube in this position. Record atmospheric pressure by reading the laboratory barometer.

PROCESSING THE DATA

- 1) Determine the pressure of the air in the tube in each of the three different positions. The pressure of the trapped air in the horizontal position is the same as the pressure indicated by the barometer. With the mercury plug pushing downward on the column of air, the pressure is the barometric pressure plus the pressure exerted by the mercury plug measured in cm or mm of Hg. With the mercury plug down the trapped air is expanded. The pressure is the barometric pressure minus the pressure exerted by the mercury plug.
- 2) Calculate the PV product for each different position of the capillary tube.
- 3) What effect would increasing the temperature have on the value for the PV product?

Experiment 11

The Reaction of Magnesium with Hydrochloric Acid

In this experiment you will determine the volume of hydrogen gas which is produced when a sample of magnesium reacts with 6 *M* hydrochloric acid. This is a solution of hydrogen chloride gas in water. The volume of the hydrogen gas produced will be measured at room temperature and pressure — conditions that matter when determining the volume of a gas. The data you obtain will enable you to answer the question: How many liters of dry hydrogen gas at room temperature and 1 atmosphere can be produced per mole of magnesium metal used?

PROCEDURE

- Obtain a piece of magnesium ribbon approximately 5 cm long. Measure the length of the ribbon carefully and record this to the nearest 0.01 cm. Your teacher will tell you the mass of 1.00 meter of the ribbon. Since it is uniform in thickness and width, you can calculate the mass of the magnesium you used.
- Look at Figure 11-1 to see how the magnesium ribbon is encased in a cage of fine copper wire. Notice the cage has no large openings through which small pieces of magnesium ribbon could escape.
- Set up a ring stand and utility clamp in position to hold a 50 ml gas measuring tube as shown in Figure 11-3. Fill a 400 ml beaker about two-thirds full of tap water. Place it near the ring stand.

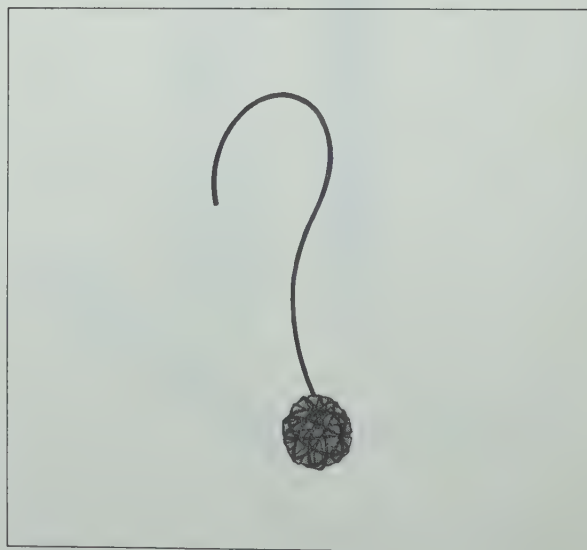


Figure 11-1

Mg ribbon wrapped in Cu wire cage.

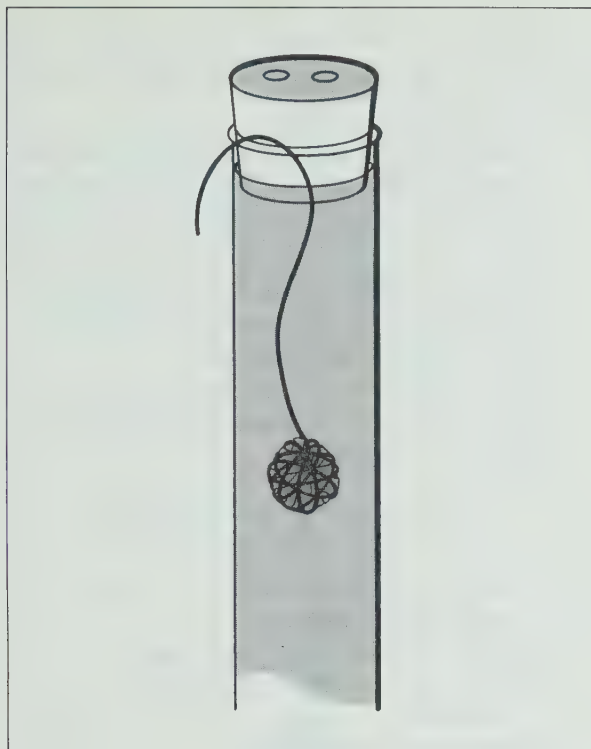


Figure 11-2

Mg in Cu cage positioned near open end of tube.

- d. Incline the gas measuring tube slightly from an upright position and pour in about 10 ml of 6 M hydrochloric acid.
- e. With the tube in the same position, slowly fill it with tap water from a beaker or bottle. While pouring, rinse down any acid that may be on the side of the tube so that the liquid in the top of the tube will contain very little acid. Try to avoid stirring up the acid layer in the bottom of the tube. Bubbles clinging to the side of the tube can be dislodged by tapping the tube gently.
- f. Holding the copper coil by the handle, insert the cage about 3 cm down into the tube. See Figure 11-2. Hook the copper wire over the edge of the tube and hold it there by inserting the rubber stopper. The tube should

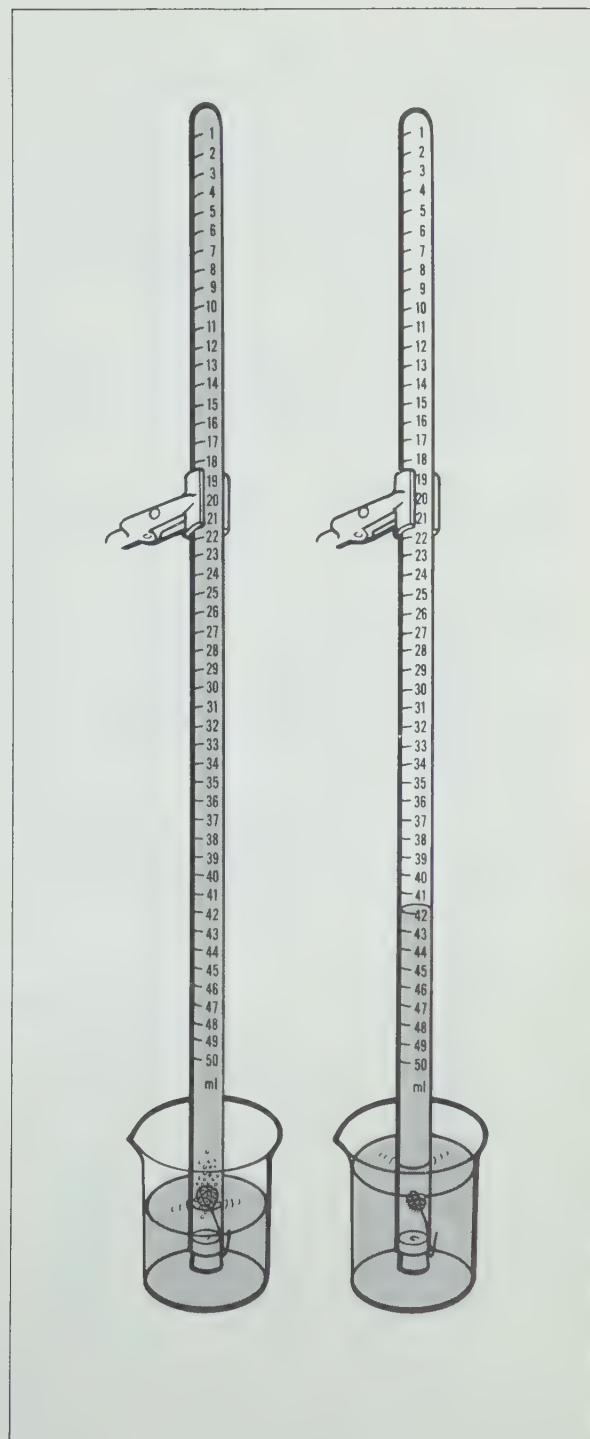


Figure 11-3

Using the gas measuring tube.

be completely filled so that the stopper displaces a little water when put in place.

- g. Cover the hole in the stopper with your finger and invert the tube in the container of water, as shown in Figure 11-3. Clamp it in place. The acid being more dense than water will fall down through it and eventually react with the metal.
- h. After the reaction stops, wait for about 5 minutes to allow the tube to come to room temperature. Dislodge any bubbles clinging to the side of the tube.
- i. Cover the hole in the stopper with your finger and transfer the tube to a large cylinder or battery jar which is almost filled with water at room temperature. See Figure 11-4. Lower or raise the tube until the level of the liquid inside the tube is the same as the level outside the tube. This permits you to measure the volume of the gases in the tube (hydrogen and water vapor) at room pressure.
Read the gas volume. Your eye should be at the same level as the bottom of the meniscus (the lens-shaped surface taken by

the water in the tube). See Figure 11-5. Record the volume of the gas to the nearest 0.01 milliliter.

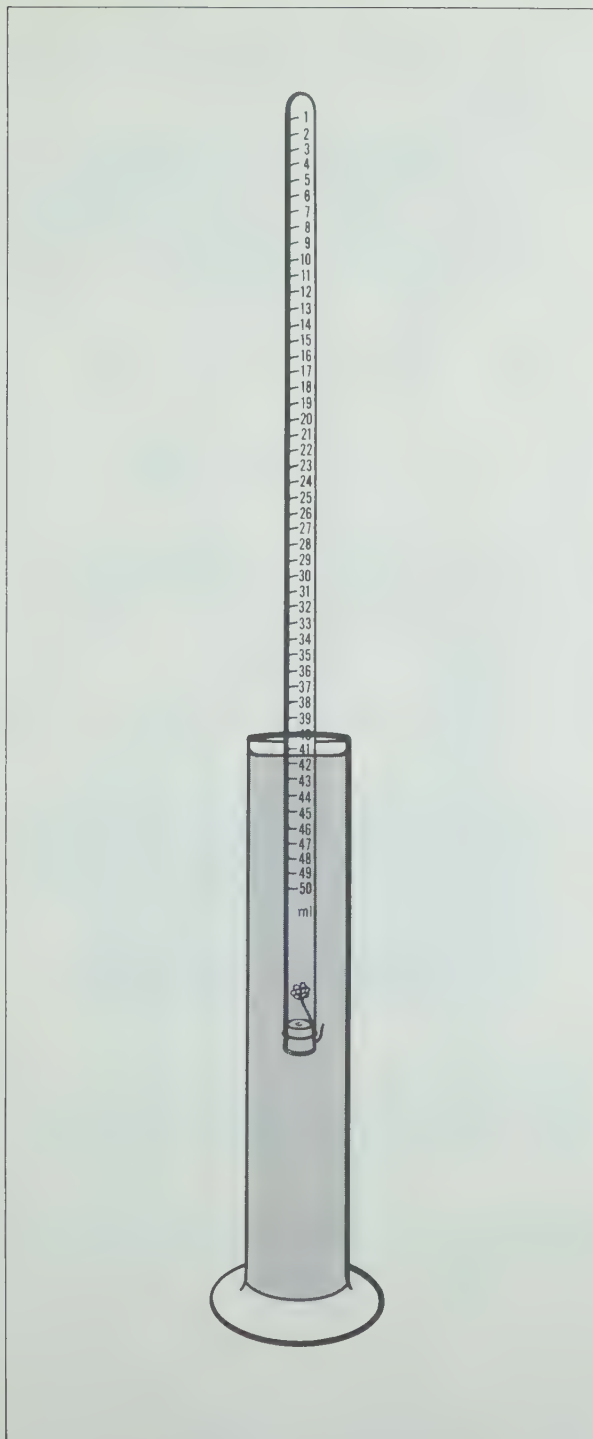
- j. Remove the gas measuring tube from the water and pour the acid solution it contains down the sink. Rinse the tube with tap water.
- k. Record the room temperature. Your teacher will give you the room pressure or will assist you in reading the barometer.
The experiment may be repeated with another sample of magnesium to check your results, if time permits.

Your Data Table Should Include the Following:

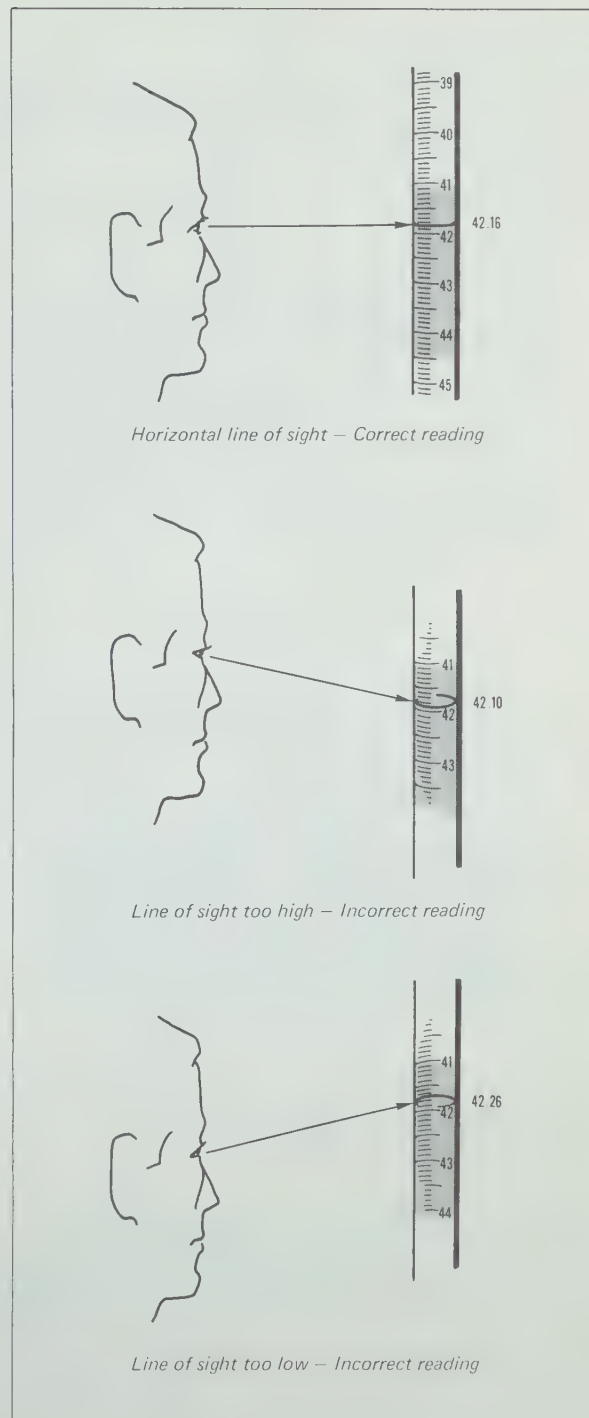
mass of magnesium ribbon in grams per meter (from teacher)
length of magnesium ribbon used
volume of hydrogen and water vapor collected
temperature of the water
temperature of the room
barometer reading (room pressure)
vapor pressure of water at the above temperature (see the following table)

Vapor Pressure of Water at Various Temperatures

Temperature (°C)	Pressure (mm Hg)	Temperature (°C)	Pressure (mm Hg)
15	12.8	23	21.0
16	13.6	24	22.4
17	14.5	25	23.7
18	15.5	26	25.2
19	16.5	27	26.7
20	17.5	28	28.3
21	18.6	29	30.0
22	19.8	30	31.8

**Figure 11-4**

Measuring the volume of gas.

**Figure 11-5**

Reduce error by reading bottom of meniscus with eye at proper level.

PROCESSING THE DATA

- 1) Determine the mass of the magnesium you used knowing the grams per meter and the length of your piece of ribbon.
- 2) Determine the number of moles of magnesium used.
- 3) Determine the partial pressure of the hydrogen gas. Since the hydrogen gas was collected over water, the gas in the tube consists of a mixture of hydrogen gas and water vapor. The total pressure caused by these two gases was made equal to room pressure in step i. See the hypothetical case illustrated in Figure 11-6A. Mathematically this can be expressed

$$p_{\text{H}_2} + p_{\text{H}_2\text{O}} = P_{\text{room}}$$

The pressure of the room may be determined by reading the barometer. The pressure of the water vapor, $p_{\text{H}_2\text{O}}$, can be obtained from the table given above. The values in the table were obtained by measuring the pressure of water vapor above liquid water at various temperatures. The partial pressure of the hydrogen can then be calculated as follows:

$$p_{\text{H}_2} = P_{\text{room}} - p_{\text{H}_2\text{O}}$$

See Figure 11-6B for illustration of the pressure due to the hydrogen alone.

- 4) Determine the volume the hydrogen gas would have at one atmosphere pressure (760 mm).

You have learned that for a given temperature the product of the pressure and volume of a gas is a constant. To calculate the new volume, V_{new} at 760 mm pressure, the following mathematical relation can be stated

$$p_{\text{H}_2} \cdot V_{\text{measured}} = 760 \cdot V_{\text{new}}$$

or

$$V_{\text{new}} = V_{\text{measured}} \times \frac{p_{\text{H}_2}}{760}$$

- 5) Calculate the volume of dry hydrogen which would be produced by 1 mole of magnesium at room temperature and 1 atmosphere pressure.
- 6) Given that 1 mole of Mg produces 1 mole of hydrogen, H_2 , what is the volume of 1 mole of hydrogen at room temperature and 1 atmosphere pressure?
- 7) If 1 mole of hydrogen weighs 2.0 g, what is the mass of a liter (the density) of hydrogen at room temperature and 1 atmosphere pressure?

ADDITIONAL INVESTIGATION

Consult your teacher before proceeding.

Determine the volume of hydrogen gas produced when a mole of another metal reacts with an acid.

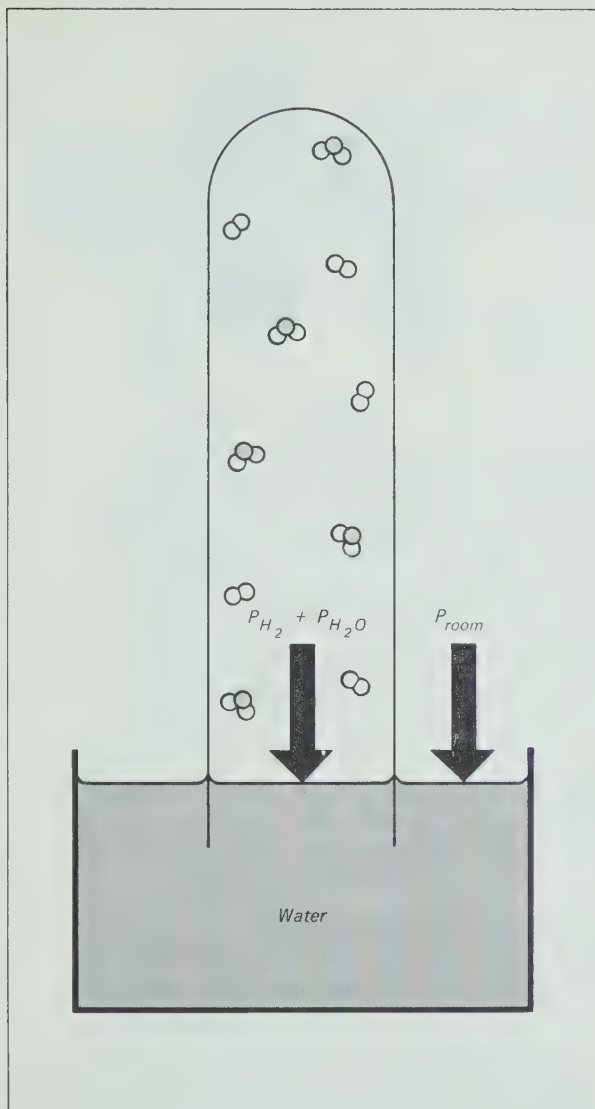


Figure 11-6A

Pressure of $H_2(g)$ and $H_2O(g)$ balanced against the pressure of the air in the room.

$$P_{H_2(g)} + P_{H_2O(g)} = P_{room}$$

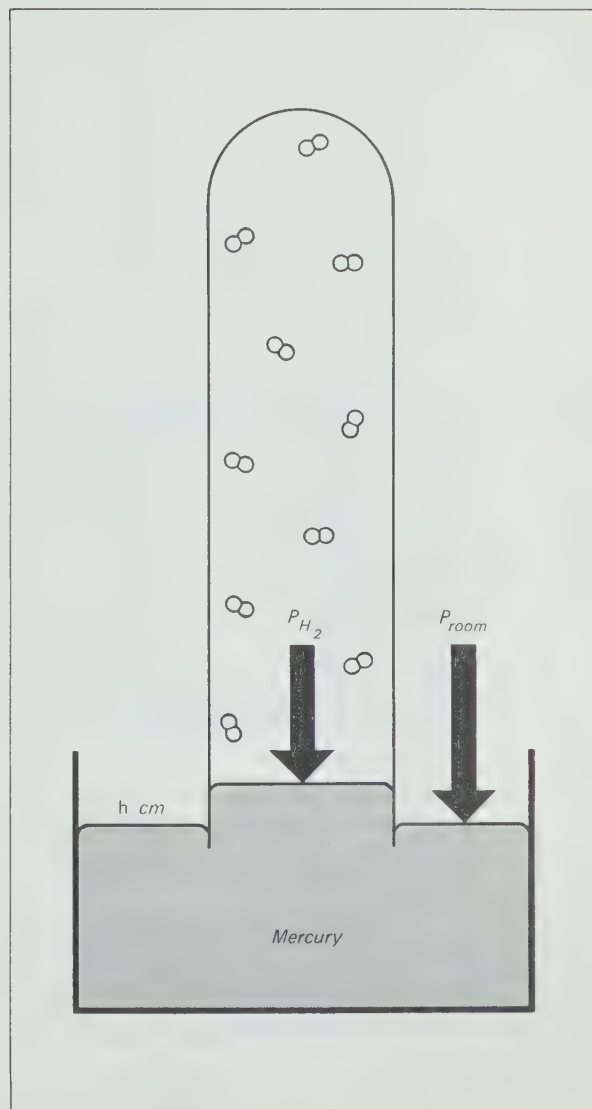


Figure 11-6B

If the water is replaced with mercury, the pressure exerted by the trapped gas will drop. The Hg will rise in the tube. Now the pressure of the air in the room is balanced by the pressure of the $H_2(g)$ plus the pressure due to the column of Hg h cm high. The vapor pressure of water is h cm of mercury.

$$P_{H_2(g)} + h = P_{room} \quad \text{or} \quad P_{H_2(g)} = P_{room} - P_{H_2O(g)}$$



Experiment 12

Warming and Cooling Behavior of a Pure Substance

Although there are only about 100 elements known to man, there are about 4 million different compounds which are made of these elements. Each year several hundred thousand new compounds are reported. Most of these substances, both elements and compounds, exist as solids at ordinary temperatures. Only a small number are liquids or gases at normal temperatures. Each has a characteristic set of properties which serves to give it identity. One of these properties is called the *melting temperature*. These characteristic temperatures have been determined for most of the known compounds and are available in the scientific literature.

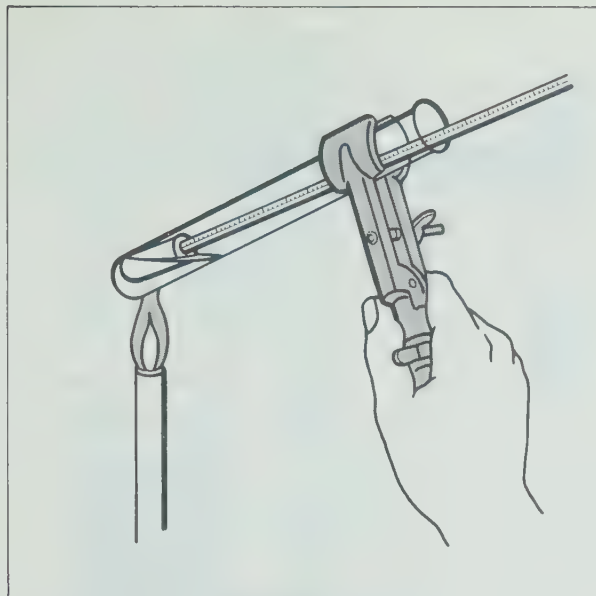
In this experiment you will study the warming and cooling behavior of at least one pure substance, *paradichlorobenzene* (para-di-chloro-benzene). This substance is a common moth repellant and can be represented by the chemical formula $C_6H_4Cl_2$.

First, solid *paradichlorobenzene* will be heated slightly above its melting temperature. Then time and temperature data will be recorded every 30 seconds as the substance is cooled. Finally, time-temperature data will be obtained as the substance is warmed.

PROCEDURE

Cooling Behavior

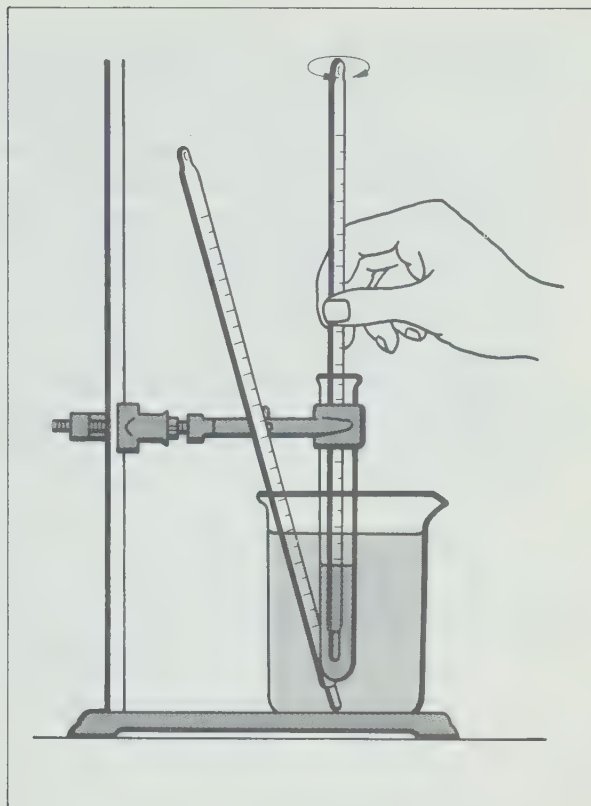
- a. In this experiment you will work with a partner; one will be the observer and the other the recorder. The recorder should prepare a data table in his notebook. Make two carbon copies of the data. The original will be handed in to your teacher at the end of the experiment, and one carbon copy is for the observer. See the sample data table at the end of the procedure section.
- b. Fill a 400 ml beaker four-fifths full of tap water. Heat the water until the temperature is between 30 and 35°C. Leave a thermometer in the water bath and place the beaker on the base of the ring stand.
- c. Place about 15 grams of *paradichlorobenzene* in a large test tube. Warm the solid by moving the test tube back and forth in the burner flame until the solid starts to melt. Place a second thermometer in the liquid and

**Figure 12-1**

Melting the *paradichlorobenzene* crystals.

continue warming until the temperature is between 65 and 70°C. Be careful not to overheat the *paradichlorobenzene*. Move the test tube back and forth in the flame as shown in Figure 12-1.

- d. Clamp the test tube in position above the beaker of water. The recorder will signal the time every 30 seconds and will record all the observations noted by the observer.
- e. When all is ready, check the temperature of the *paradichlorobenzene* with one thermometer and the temperature of the water bath with another. On signal from the recorder, immediately immerse the test tube into the water bath. The water level outside the test tube should be above the level of the liquid inside the test tube. Clamp the test tube in position as shown in Figure 12-2.
- f. Stir the liquid *paradichlorobenzene* continuously with the thermometer as long as any liquid remains. Don't let the thermometer become stuck in the solid.
- g. Record the temperature of the *paradichlorobenzene* each 30 seconds and the tempera-

**Figure 12-2**

Apparatus for observing the cooling behavior.

ture of the water bath each minute until the temperature of the solid drops below 40°C. Be sure your observations include a note of when solidification begins and when it is completed.

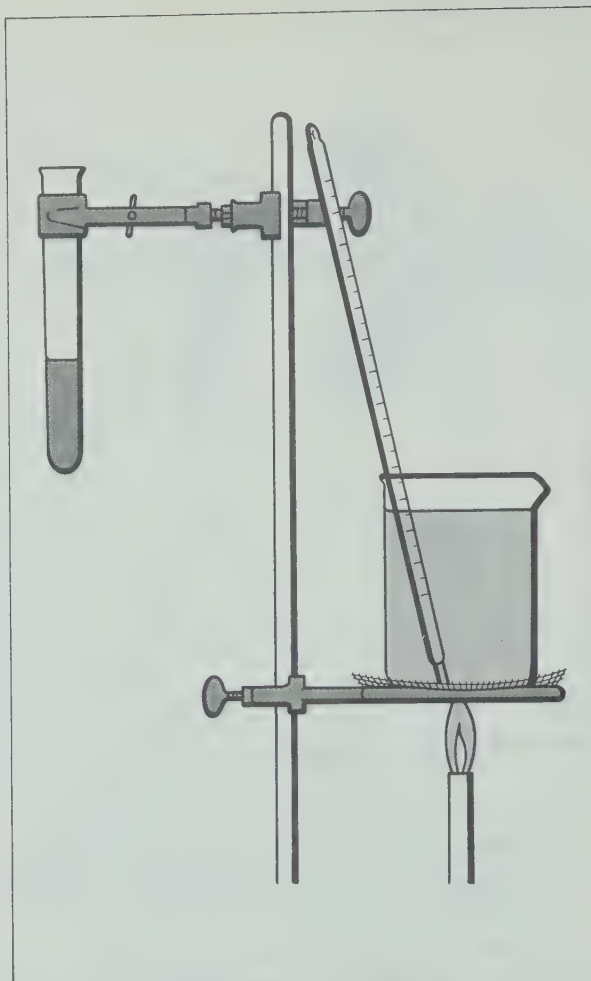
Warming Behavior

- h. Partners should exchange duties at this point. Raise the test tube out of the water bath and turn the clamp around so the test tube is on the other side of the ring stand, opposite the water bath. See Figure 12-3. Place the beaker, four-fifths full of water, on a wire gauze on a ring. Heat the water until the temperature is between 65 and 70°C.
- i. Record the temperature of the solidified *paradichlorobenzene* with one thermometer and the temperature of the hot water bath

with another. On signal from the recorder, immerse the test tube into the water bath. The water level outside the test tube should be above the level of the solid inside the test tube. Record the temperature of the *paradichlorobenzene* each 30 seconds and the temperature of the water bath each minute. Stir the *paradichlorobenzene* as soon as the solid comes free from the walls of the test tube. Continue to stir and record temperatures until the solid has melted and the temperature of the liquid *paradichlorobenzene* rises to about 60°C. Note the time when melting begins and when it is completed.

Figure 12-3

Apparatus for observing warming behavior.



Sample Data Table

Time (minutes)	Cooling Data			Warming Data		
	Temperature, °C		Observations	Temperature, °C		Observations
	PDCB	Water		PDCB	Water	
0						
1/2						
1						
1 1/2						
2						
2 1/2						
3						
3 1/2						
4						
4 1/2						
etc.						

PROCESSING THE DATA

- 1) In the evaluation of this experiment it helps to look at the data in graphical form. Use a full page of graph paper. Present time along the horizontal axis and temperature along the vertical axis. Plot warming and cooling data on the same graph. Also plot the water bath data on the same graph. Draw smooth curves to represent the warming and cooling behavior of the *paradichlorobenzene*. Draw dotted smooth curves for the water bath data. Be sure to identify each curve.
- 2) Write a report summarizing the results of this experiment. Compare the shape of the water bath curves to the shape of the *paradichlorobenzene* warming and cooling curves. Give an explanation to account for any observed differences. Include in the report your interpretation of the shapes of these curves.

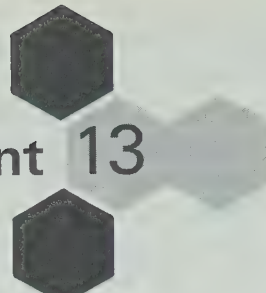
- 3) What effect would increasing the amount of *paradichlorobenzene* have on the shape of the curves?
- 4) From your data, what is the melting temperature for *paradichlorobenzene*?
- 5) From your data, what is the freezing temperature for *paradichlorobenzene*?

A QUESTION TO WONDER ABOUT

Why do the warming and cooling curves have the characteristic shape shown by your graph?

ADDITIONAL INVESTIGATION

Determine the cooling behavior of some other pure substance. Your teacher will suggest suitable substances.



Experiment 13

Energy Needed to Melt Ice

Pure substances have characteristic melting and freezing behavior. Pure water changes from a solid (ice) at 0°C to a liquid at 0°C as energy is added to it.

In this experiment you will determine the energy required to melt one gram of ice by letting an excess of it interact with warm water in a Styrofoam cup (ice calorimeter). The ice will cool the water to about 0°C . The energy given up by the water as it cools is the energy used to melt the ice. Recall that one calorie is required to change the temperature of one gram of liquid water one Celsius degree.

PROCEDURE

- Warm about 125 ml of water to about 50°C .
- Measure 100 ± 1 ml of this warm water into a Styrofoam cup. Record the temperature of the warm water to the nearest 0.1°C .
- Obtain several ice cubes. Shake excess water from them. Place the ice in the warm water and stir the mixture until the temperature is about 0°C . Add more ice if needed to cool the water. Record the lowest temperature reached.
- After the water and ice mixture has cooled to about 0°C , remove the unmelted ice (using tongs). Be sure to drain back as much water as possible into the cup when you remove the ice.
- Measure the volume of water remaining in the calorimeter to the nearest milliliter.

PROCESSING THE DATA

- Determine the mass of ice melted (the density of water is one gram per milliliter).
- Determine the change in temperature of water, Δt .
- Calculate the energy released by 100 grams of liquid water as it is cooled through Δt .
- Calculate the calories of energy required to melt one gram of ice.
- Using the results of this experiment, determine the number of calories required to melt one mole of ice.
- Write an equation for the fusion (melting) of ice, and include the energy term in kcal/mole of water.



Experiment 14

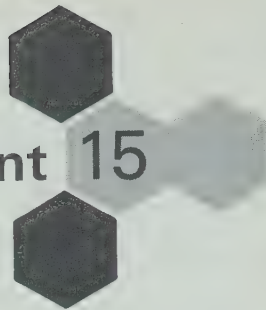
Energy of Crystallization

You have observed that energy must be added to change a substance from a solid to a liquid. Where does the energy go? Why does the temperature remain constant during a change of phase? Why does the temperature stay constant even though energy is removed during the change from liquid to solid? Perhaps this experiment will help you find answers to some of these questions.

You will melt a substance, sodium thiosulfate pentahydrate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, and cool it below its freezing temperature. Many substances tend to *undercool*, that is, cool below their freezing temperatures without freezing or crystallizing. Sodium thiosulfate pentahydrate is a good example of a substance which undercools. Crystallization can be induced by *seeding* the liquid with a small crystal of the substance.

PROCEDURE

- a. Weigh a large test tube. Add about 20 grams of solid sodium thiosulfate pentahydrate and determine the mass to the nearest 0.1 gram.
- b. Melt the sodium thiosulfate using a boiling water bath. Continue to warm the melted sodium thiosulfate to about 70°C . Leave the thermometer in the test tube.
- c. Transfer the test tube to a cold water bath. The temperature of the water in the bath should be about room temperature. Allow the liquid sodium thiosulfate to cool below 35°C . Do not stir the liquid.
- d. After the liquid has cooled, remove it from the cooling bath. Drop a small crystal of sodium thiosulfate into the test tube and observe what happens. Record observations in your notebook.
- e. Think of an experiment you can do to measure the energy released as liquid sodium thiosulfate changes to solid. Make an outline of your proposed procedure and discuss it with your teacher before you proceed. Save the sodium thiosulfate. You will need it again in your experiment.



Experiment 15

Cooling Behavior of a Solution

In this experiment you will observe the cooling behavior of a solution. The solvent to be used is acetamide, CH_3CONH_2 . The substance to be dissolved (solute) will be assigned from the following list:

urea	60 g/mole
camphor	152 g/mole
sodium iodide	160 g/mole
potassium iodide	166 g/mole

PROCEDURE

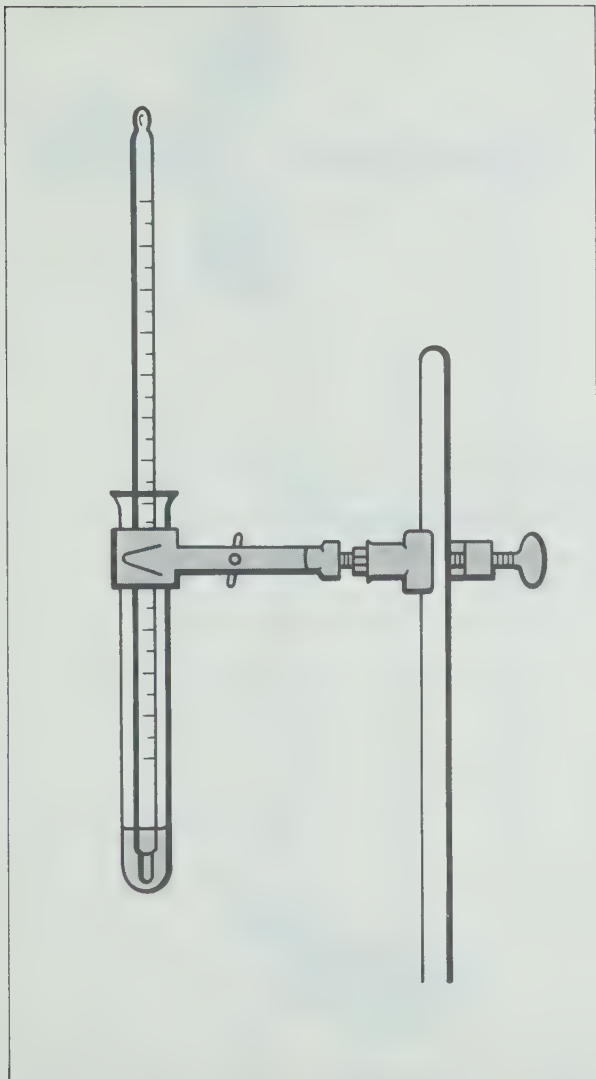
- Melt 5.0 grams of acetamide in a clean dry 18×150 mm test tube. Melt the solid by carefully warming the test tube directly in a burner flame. Do not overheat the liquid. Continue to warm the acetamide until the temperature reaches about 90°C .
- Clamp the test tube in position as shown in Figure 15-1. Obtain time and temperature data at 30 second intervals as the acetamide cools in air. Stir the liquid continuously using a thermometer, until a constant temperature has been reached during solidification.
- Your teacher will assign you one of the compounds listed above. Add 0.0025 mole of the assigned solute to the 5.0 grams of acetamide used in step a.
- Melt the mixture and heat the solution to about 90°C . Allow the solution to cool in air as you did in step b. Obtain time and temperature data at 30 second intervals as the solution cools. Continue to stir the mixture and collect data for 3 or 4 minutes after freezing begins.

- To the mixture from step d, add an additional 0.0025 mole of the same solute used in step c.

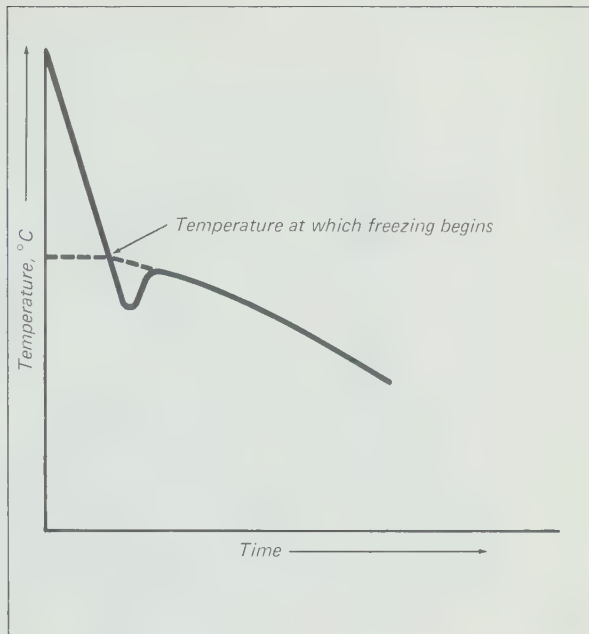
PROCESSING THE DATA

- Plot, on the same graph, the time and temperature data from all three determinations. Present temperature along the vertical axis and time along the horizontal axis. Choose appropriate scales. Label each curve. For the solutions, indicate how much of the assigned solute was dissolved in acetamide. Sometimes these solutions tend to under-cool. That is, they cool to a temperature below the freezing temperature before crystal formation begins. When crystallization begins, the temperature suddenly rises. This sudden change in temperature is due to the release of thermal energy. The freezing temperature is obtained by extrapolation as shown in Figure 15-2.
- Determine the change in freezing temperature, Δt_f , caused by adding 0.0025 mole of solute to acetamide.

*acetamide
5.0 g = 0.025 moles
0.0025 moles*

**Figure 15-1**

Apparatus for cooling acetamide in air.

**Figure 15-2**

Extrapolation to determine the freezing temperature of a solution or pure substance after undercooling.

- 3) Determine the additional change in freezing temperature, Δt_f , when the second amount of solute was added.
- 4) Average the Δt_f values for each 0.0025 mole of solute added.

- 5) Compare average values of Δt_f when 0.0025 mole of each solute was added to 5.0 grams of acetamide. Search for a regularity among these data.
- 6) The temperature remained essentially constant during the solidification of acetamide. During the solidification of the solutions the temperature dropped. Explain.

ADDITIONAL INVESTIGATION

Predict how much the freezing temperature for acetamide will be lowered by the addition of 0.0025 mole of anhydrous CaCl_2 . After making your prediction do an experiment to check it out.

Experiment 16

Formula of a Precipitate

In this experiment, you will prepare 0.50 *M* solutions of lead nitrate and sodium iodide [$\text{Pb}(\text{NO}_3)_2$ and NaI]. A precipitate is formed when these solutions are mixed. You will study the mole relation between reactants and products. Moles of reactants will be determined from volume and concentration of solutions used. Moles of precipitate will be determined from its mass.

PROCEDURE

Part I. Preparation of Solutions

To reduce duplication of effort, you will work with a partner. Each partner will prepare *one* of the 0.50 *M* solutions, which will be shared as the remainder of the experiment is performed.

- Before coming to the laboratory, each partner should calculate the mass of both compounds needed to make 50.0 ml of a 0.50 *M* solution of each. Check your answers with those of your partner before proceeding to step b.
- In the laboratory, each partner will weigh to the nearest centigram the calculated amount of one solid. First determine and record the mass of a 100 ml beaker. Set the balance for the mass of the beaker plus the mass of the solid needed. Figure 16-1 shows the technique for transferring solid from a bottle. Gently roll the bottle to slowly transfer solid from bottle to beaker. If too much is transferred, discard the excess. Do not return it to the bottle. Clean up all spilled chemicals.

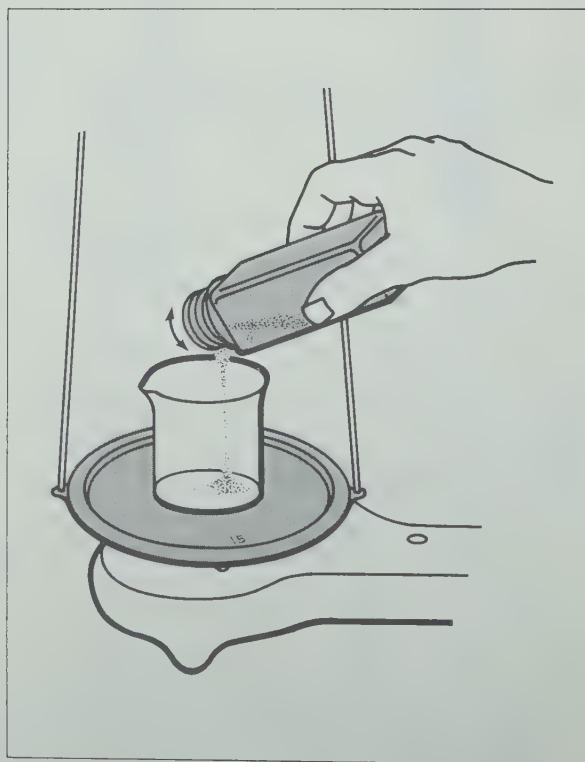
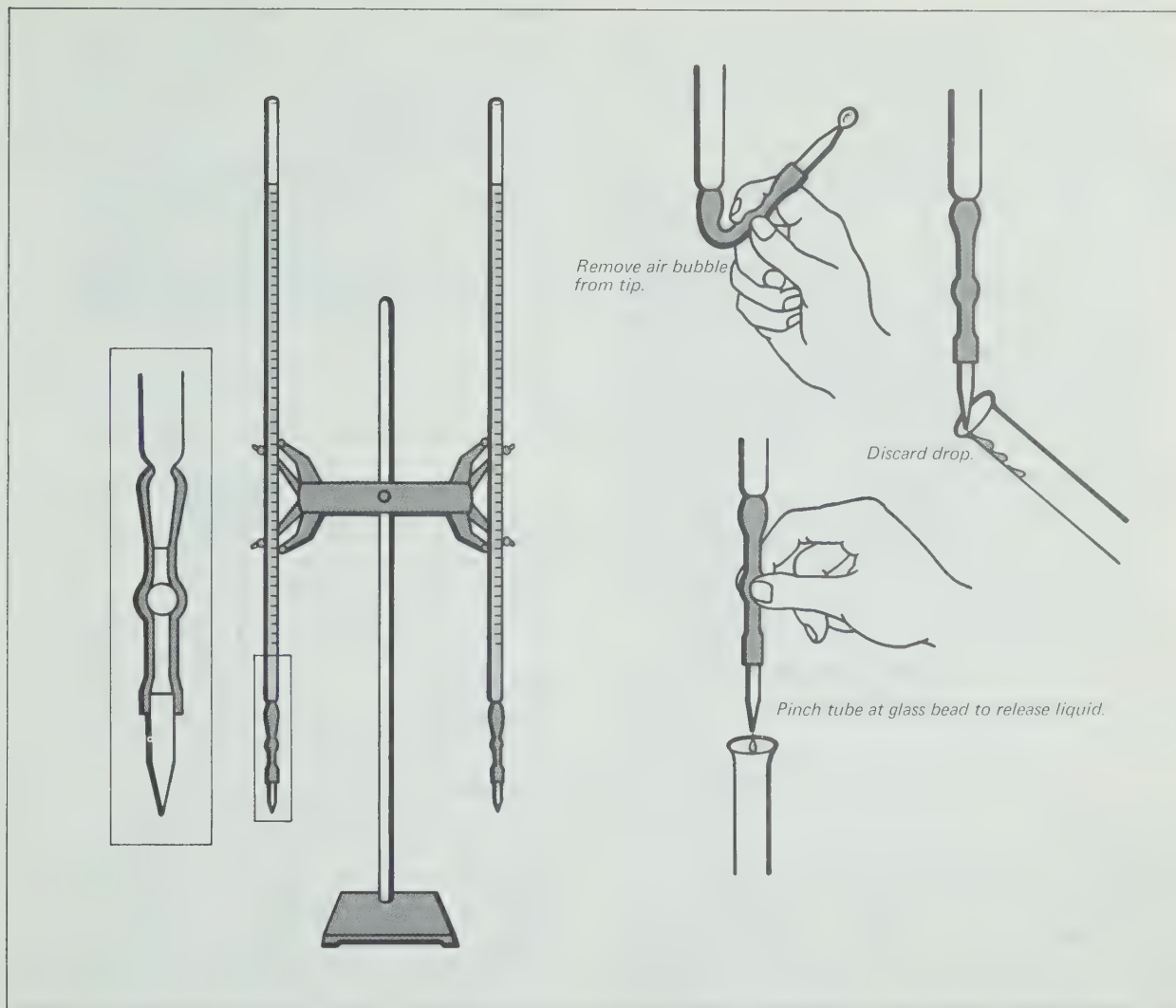


Figure 16-1

Removing solid from bottle.

**Figure 16-2**

Buret assembly and use.

- c. To dissolve the solid, measure 40 ml of distilled water into the beaker. While the solid is dissolving, proceed with step d, but occasionally swirl the solution.
- d. Assemble a buret as shown in Figure 16-2. Be sure the buret is clean, well rinsed with distilled water, and labeled.
- e. If the solid has not yet dissolved, heat the beaker gently and swirl it until the solid disappears.
- f. Carefully pour your solution into a clean 50 ml graduated cylinder. Rinse the beaker with 4 ml of distilled water. Add this rinse solution to the cylinder. Repeat. Finish with a total of 50.0 ml of solution in the cylinder. Pour the solution into a clean dry labeled beaker and mix well.
- g. Use 3 ml of the solution to rinse thoroughly all inside surfaces of the buret. Discard the rinsing solution. Fill the buret with your

solution to slightly above the zero mark. Allow some solution to run out to eliminate air from the tip and leave the tip full of solution. See Figure 16-2.

Part II. Mixing Predetermined Volumes of the Two Solutions

- h. Prepare eight clean 13×100 mm test tubes. The tubes should be numbered one through eight in Roman numbers. One partner should prepare the first four, the other the last four. Add to these tubes carefully measured volumes of the solutions you and your partner prepared. The volumes needed are shown in the following table. When adding the second solution, add a few drops and shake the test tube to mix the solutions. Add a few more drops and shake the tube. Add half the remaining solution and shake well. Add the rest of the solution and shake the tube.

In your notes, record the buret readings before and after adding each solution to the numbered test tubes.

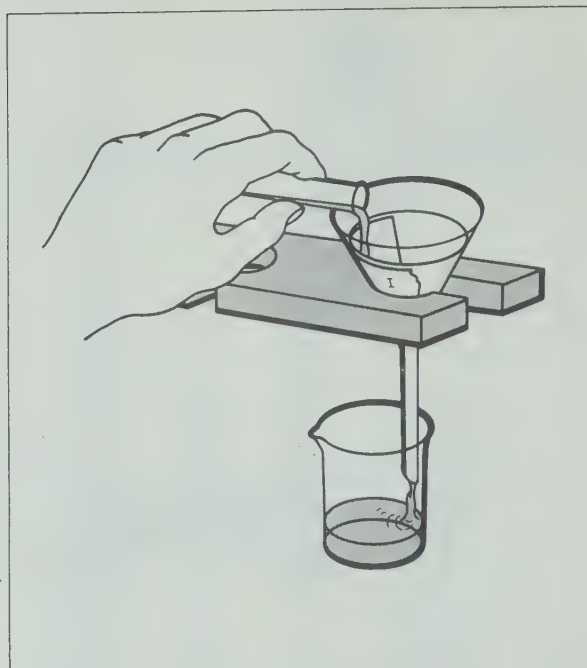


Figure 16-3

Apparatus for filtering.

Milliliters of Solutions Used

Tube Number	I	II	III	IV	V	VI	VII	VIII
0.50 M $\text{Pb}(\text{NO}_3)_2$	1.00	2.00	3.00	4.00	5.00	6.00	7.00	8.00
0.50 M NaI	8.00	7.00	6.00	5.00	4.00	3.00	2.00	1.00

- i. Shake the tubes well. Let them stand in a beaker in your locker until next lab period.
- j. Take the tubes out gently and arrange them in numerical order. Note the heights of precipitate in each tube. The precipitate is lead iodide.

Part III. A Quantitative Study of the Amount of Precipitate Formed

You can perhaps think of several reasons to explain why the heights of the precipitates formed in Part II do not give a precise measure of the amount of precipitate present. You can overcome these difficulties by determining the mass of precipitate formed in each tube. Since this is a rather

small amount, a small absolute error in weighing will be a large percent error. The percent error can be reduced by combining precipitates from several experiments.

- k. Four pairs of students should now form a group of eight. Each of these eight students will be responsible for four tubes, each of which has the same number. One student collects four tubes numbered I, another student collects four tubes numbered II, etc.
- l. Each student will filter the contents of four tubes, all having the same Roman number. To do this, set up a funnel as shown in Figure 16-3. Obtain a piece of filter paper of known mass. Record the mass. Number

the filter paper with the number of your tubes. Carefully pour the contents of each tube into the filter paper. Rinse each tube with filtrate until essentially all solid is transferred into the filter cone. Collect the filtrate in a beaker. Wash the precipitate once with a few ml of cold water.

- m. Put the filter paper in a numbered beaker and your teacher will tell you how to dry the precipitate.
- n. Your teacher will tell you how to dispose of the filtrate.
- o. Determine the mass of the dried precipitate.

PROCESSING THE DATA

- 1) Obtain the mass of the dried precipitate from each member of your group. From this, determine the average grams of lead iodide in each tube. Be sure the mass of the filter paper is not included.
- 2) Determine the number of millimoles of $\text{Pb}(\text{NO}_3)_2$ and of NaI added to each tube.
- 3) Present your results in graphical form. Show millimoles of $\text{Pb}(\text{NO}_3)_2$ along the horizontal axis starting at the origin. Show millimoles of NaI along the same horizontal axis but finishing at the origin. As the third label for this axis, show the tube number. See Figure 16-4. Show grams of lead iodide along the vertical axis. Label the graph and each axis.

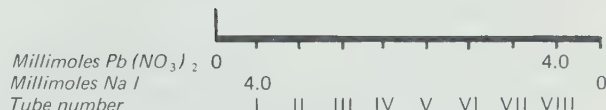


Figure 16-4

Horizontal axis for graph.

- 4) How many millimoles of Pb^{2+} were available when the greatest amount of lead iodide was formed?
- 5) How many millimoles of I^- were available when the greatest amount of lead iodide was formed?
- 6) What is the simplest whole number ratio of $\frac{\text{moles I}^-}{\text{moles Pb}^{2+}}$ when the greatest amount of lead iodide was formed?
- 7) What is the formula of lead iodide?
- 8) Why was the same amount of lead iodide not formed in each tube?
- 9) According to this experiment, when the amount of lead iodide formed was at a maximum, how many moles of lead iodide were formed for each 1.00 mole of $\text{Pb}(\text{NO}_3)_2$ used?
- 10) Write a balanced chemical equation for the formation of lead iodide from ions.

Experiment 17

Reactions Between Ions in Aqueous Solution

Ionic solids dissolve in water to form aqueous solutions which conduct electricity. These solutions contain both positive and negative ions in such numbers that their net electric charge is zero.

In this experiment you will mix various ionic solutions, two at a time, to determine which combinations form precipitates. Knowing which ions are present makes it possible to deduce which of the possible ion combinations are responsible for the precipitates.

Determine the number of possible combinations you can make using six different solutions combined two at a time. For example, three different solutions combined two at a time provide six possible combinations:

1 and 1	2 and 2
1 and 2	2 and 3
1 and 3	3 and 3

There is no need to combine 1 with 1, 2 with 2, and 3 with 3. Thus only three combinations need to be tested. Make a similar analysis combining six different solutions. Organize a table for your observations. Your teacher will provide you with a set of six solutions. List the numbers for the solutions as shown in the sample table for three solutions combined two at a time. Include in the table the formulas for the ions present in each of your solutions. The solutions shown in the sample table are not necessarily the solutions you will be using.

Sample Table for Three Different Solutions
Combined Two at a Time

		3	2	1
		$\text{Ag}^+, \text{NO}_3^-$	K^+, Cl^-	$\text{Ba}^{2+}, \text{NO}_3^-$
1	Ba^{2+} NO_3^-			
2	K^+ Cl^-			
3	Ag^+ NO_3^-			

If you do not know the charge associated with each ion, refer to Appendix 2 which gives this information.

PROCEDURE

- a. Place a drop or two of one of your solutions on a clean piece of plastic film. Add a drop or two of a second solution to it. For each succeeding test choose a clean spot on the plastic film.
- b. Continue testing pairs of solutions until all possible combinations have been tested.
- c. Make an entry in your data table to indicate those combinations in which a precipitate is formed. A precipitate will appear as a cloudy or milky mixture in the combined drops.
- d. If so directed by your teacher, you may wish to repeat the above procedure using another set of solutions.

PROCESSING THE DATA

- 1) Since the pairs of ions listed for each solution were present in the solution, you may assume that each precipitate formed was due to a new combination of these ions. For example, if you mix aqueous solutions of AgNO_3 and NaCl there are two new combinations of ions possible. The silver nitrate solution contains $\text{Ag}^+(\text{aq})$ and $\text{NO}_3^-(\text{aq})$. The sodium chloride solution contains $\text{Na}^+(\text{aq})$ and $\text{Cl}^-(\text{aq})$. Possible new combinations of these ions are AgCl and NaNO_3 .

Make a list of all possible new combinations of ions for each pair of solutions that produced a precipitate.

- 2) Examine your data and propose explanations that will account for the fact that a precipitate was formed in some combinations and not in others.
- 3) Examine your data and see if you can justify eliminating some of the combinations listed as possible precipitates.
- 4) Write equations to indicate what you consider to have happened in each case in which there was a precipitate formed. Use ions to represent the species in the reacting solutions, but for those products that were precipitates write a formula for the compound. Place (aq) after those species in solution, and (s) after the precipitates. Be sure to write the equations so that both atoms and charge are conserved. For example:
$$\text{Ag}^+(\text{aq}) + \text{NO}_3^-(\text{aq}) + \text{Na}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \longrightarrow \text{AgCl}(\text{s}) + \text{Na}^+(\text{aq}) + \text{NO}_3^-(\text{aq})$$
- 5) Rewrite these equations leaving out the ions not involved in the reaction. Such an equation, which shows only the predominant reacting species, is called a *net ionic equation*.



Experiment 18

Types of Solutions

In this experiment you will investigate unsaturated, saturated, and supersaturated solutions. The solute used is hydrated sodium acetate, $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3 \text{H}_2\text{O}$ which we will shorten to NaAc.

PROCEDURE

- a. To 5 ml of water in an 18×150 mm test tube add 3 crystals of NaAc. Shake it until the salt dissolves. Add 3 more crystals and shake. Did they dissolve? If so, was the solution saturated at first? Is it now?
- b. To the solution made in step a, add a teaspoonful of NaAc. Shake the tube for at least one minute. Note any temperature change. If all the crystals dissolve, add a half teaspoonful and shake the tube one minute. Decant the clear solution into a dry test tube. Add one crystal to the decanted liquid and shake one minute. Did this crystal dissolve? If not, is this solution now saturated?
- c. Pour the solution back on the crystals, add two more teaspoonfuls of crystals. Heat the tube until the crystals dissolve. While heating, gently shake the tube to mix the contents thoroughly.
- d. Stand the test tube in a beaker of cool water for about 5 minutes. Do not disturb it during this time. If crystals appear in the solution, heat it until they dissolve and again stand the test tube in cool water.
- e. After the solution has cooled to about room temperature, hold the test tube in your hand so you can note the temperature of the solution. Add one crystal of NaAc.
- f. Dispose of the chemicals as directed by your teacher.

PROCESSING THE DATA

- 1) What experiment would you do at the end of step c to determine if the solution produced was saturated? Explain why you think it was or was not saturated.
- 2) Because of what happened in step e, do you think the solution produced in step d can be adequately described as saturated? Explain.
- 3) Explain the observed temperature changes as NaAc dissolved and as it crystallized out of solution.
- 4) List four *different* ways of making a saturated solution.



Experiment 19

Introduction to Qualitative Analysis

Qualitative analysis in chemistry is very much like a detective game. The characters in a detective story have methods of operation and other distinguishing features. These characteristics make it possible to identify individuals as having been responsible for certain acts. The clues that one observes are evidence of some kind of interaction. In qualitative analysis you will make use of clues, evidence of chemical interaction, to help you identify the presence of specific ions in solution. However, before you can expect to identify the presence of ions, you must first become familiar with their characteristic behavior (methods of operation).

In this experiment you will be given four solutions, labeled 1, 2, 3, and 4. You will discover how they behave when they are mixed with three reagents labeled *A*, *B*, and *C*. By making careful observations you will detect evidence of chemical reaction that will be characteristic for each of the solutions. These clues may be the formation of precipitates, change in color, production of a gas, or other evidence of chemical reaction.

Recording your observations in tabular form will help you recognize characteristic behavior for each of the four solutions.

The solutions and reagents used in this experiment are deliberately identified by letter or number. This will permit you to focus your attention on the reasoning involved in developing an analytical scheme rather than on the actual reaction taking place. The scheme will permit you to identify unknown solutions according to their differentiating properties.

Consider the following hypothetical case. Reagents, *X*, *Y*, and *Z*, were allowed to react with a few drops of each of the solutions I, II, III, and IV. The observations were recorded in the table as shown.

Solutions	Reagents		
	<i>X</i>	<i>Y</i>	<i>Z</i>
I	—	+ (yellow)	+ (yellow)
II	+ (white)	—	+ (yellow)
III	—	—	+ (blue)
IV	—	+ (yellow)	—

The plus (+) means distinctive evidence of reaction was observed and a minus (−) indicates no observable evidence of reaction. The color of precipitates or changes in color of solutions are also noted. If you were given an unknown solution which you were told was like one of the four solutions tested, what method would you outline to identify the unknown? How many tests would you have to use? If an unknown gives a − test with *X* and a + test with *Y*, will this identify it? Suppose another unknown gives a + test with *X*; is this sufficient evidence to identify it as one of the four solutions?

PROCEDURE

- a. Organize a table comparable to that shown. Obtain a piece of plastic film and a set of solutions and reagents.
- b. Place a drop or two of the solutions on a clean piece of plastic film. Add a drop or two of one of the reagents. Continue making drop tests on each of the solutions 1, 2, 3, and 4 using reagents *A*, *B*, and *C*, until you have tested all possible combinations. Record your results as + or − and include a description of any characteristic property for the + tests.
- c. Study the data carefully and note the identifying clues. Obtain an unknown from your teacher. Test the unknown to determine which solution it is like. Report your analysis together with a summary of the supporting evidence.
- d. Time permitting, obtain another unknown which contains a mixture of two or more of the solutions. You may assume the solutions 1, 2, 3, and 4 do not react with each other. Report your analysis together with a summary of the supporting evidence.

Experiment 20

Qualitative Analysis – Relative Solubilities

The elements of the second column of the Periodic Table all form ions with a $2+$ charge. The chemistry of these elements is so similar that they are difficult to separate. Many of their compounds are only slightly soluble. It is possible, by choosing the proper anion, to find differences in solubility which will permit you to separate the cations of these metals.

In this experiment you will study the effect of adding reagents containing selected anions to solutions containing the cations of the metals of the second column. After a systematic study of the relative solubilities of their carbonates, chromates, sulfates, oxalates, and hydroxides, you should be able to make a qualitative analysis of an unknown solution containing one or more of these cations.

cations used		anions used	
Ba^{2+}	barium	CO_3^{2-}	carbonate
Sr^{2+}	strontium	CrO_4^{2-}	chromate
Ca^{2+}	calcium	$\text{C}_2\text{O}_4^{2-}$	oxalate
Mg^{2+}	magnesium	SO_4^{2-}	sulfate
		OH^-	hydroxide

PROCEDURE

Organize a table in which you can record your observations obtained when each solution containing a cation of the second column is tested with each of the reagents listed below.

Solutions	Cations present
0.1 M $\text{Ba}(\text{NO}_3)_2$	Ba^{2+}
0.1 M $\text{Sr}(\text{NO}_3)_2$	Sr^{2+}
0.1 M $\text{Ca}(\text{NO}_3)_2$	Ca^{2+}
0.1 M $\text{Mg}(\text{NO}_3)_2$	Mg^{2+}

Reagents	Anions present
2 M $(\text{NH}_4)_2\text{CO}_3$	CO_3^{2-}
0.5 M K_2CrO_4	CrO_4^{2-}
0.2 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$	$\text{C}_2\text{O}_4^{2-}$
1 M $(\text{NH}_4)_2\text{SO}_4$	SO_4^{2-}
6 M $\text{NH}_3(\text{aq})$	OH^-

a. Draw a pattern or grid on a piece of paper to aid you in testing each of the solutions with each of the reagents. Cover the grid with a

piece of plastic film. On the film, place drops of each solution. To these drops, add a drop of reagent so that all solutions are tested with each of the reagents.

- b. If a precipitate formed with a carbonate anion, make a larger sample by adding one ml of the appropriate solution to one ml of the appropriate reagent in a 13×100 mm test tube. Heat the test tube in a boiling water bath to aid precipitation. Cool the test tube and allow the precipitate to settle. Use a centrifuge if available. Decant the liquid and discard it. Add enough 6 *M* HCl, a drop at a time, to the precipitate until no more reaction occurs. Use these samples for flame tests.

Flame Test — An Additional Confirmatory Test

- c. Obtain a flame testing wire (platinum or nichrome). Clean the wire by heating it to incandescence in the hottest part of the flame of the burner and then dip the heated wire into about 5 ml of 12 *M* HCl in a test tube. Heat the wire again after you have dipped it into the concentrated HCl (chlorides of metals are readily volatilized). Traces of sodium cause a flash of yellow which may be difficult to eliminate. Dip the clean wire into the test solution prepared in step b and then heat the wire in the burner flame. Note the characteristic flame color for each of the solutions tested. Clean the wire as directed above before you dip it in a different solution. Use the

flame test on a solution containing a mixture of the cations. In what order do the flame colors appear?

- d. Study the data table carefully. Then obtain an unknown solution containing one of the cations of the second column. Test it to determine if it contains Mg^{2+} , Ca^{2+} , Sr^{2+} , or Ba^{2+} . Report your analysis and a summary of the evidence which supports it.
- e. Optional. Secure another unknown which contains a mixture of two or more of the cations of the second column. Outline a series of sequential tests and report the result to your teacher. Include the supporting evidence.

PROCESSING THE DATA

- 1) a. Which carbonate of the second column metals has the greatest solubility? Which ones have similar solubility?
b. Describe a possible separation of the cations based upon the above differences.
- 2) a. Which chromate of the metals is the least soluble?
b. How can this difference in solubility be used in an analytical separation of a solution containing 0.1 *M* solutions of Sr^{2+} and Ba^{2+} ?
- 3) With which of the anions does the Mg^{2+} ion have the lowest solubility?
- 4) Which oxalate of these metals is the most soluble?



Experiment 21

Construction of a Logical Model

In Chapter 1 of the textbook your attention was focused on the basic activities of science :

- to gather information through observation,
- to organize this information and to seek regularities in it,
- to wonder why regularities exist, and
- to communicate the findings to others.

Up to this time in the course, you have been generally concerned with all these activities. However in Chapters 8–10, greater emphasis will be placed on item three: to wonder why regularities exist. Current theoretical explanations and models will be presented in order that you can see how scientists account for the observed regularities and principles you have studied earlier. These explanations involve atoms, electrons, and the molecules formed by chemical bonds between atoms. None of these can be directly observed. Any properties assigned to them are, therefore, interpretations of experiments providing observable results.

An analogous situation might be the attempt to determine the nature of an object contained inside a sealed box by making a series of external observations. In this experiment you will be given a sealed system which contains an object or objects. Without opening the system, you will make all the observations you can by carefully shaking, tilting, or otherwise manipulating it. Obtain data which will enable you to make closer and closer approximations of the size, shape, and physical properties of the object. The goal is not to guess what actual object the box contains, but to describe what it must be like in order to account for the behavior you have observed.

Record the observations made each time a given manipulation of the system has been performed. Make inferences based on these observations. Test your inferences by devising other experiments. Use these observations to strengthen or modify your model. Repeat this process until your model of the object is as detailed as you can make it.

List some additional experiments you would like to perform but for which the necessary equipment is not available to you. What information would you hope to get from these experiments?



Experiment 22

Qualitative Analysis: Ag^+ , Hg_2^{2+} , and Pb^{2+}

Most common metal ions form soluble chlorides except Ag^+ , Hg_2^{2+} , and Pb^{2+} . Because of this, it is possible to separate these ions from other cations as chloride precipitates. Identification can then be made on the basis of distinguishing reactions of each chloride.

In the first part of this experiment you will become familiar with a few reactions used to identify the silver, mercurous, and lead ions in aqueous solution. From these observations you should be able to devise a method by which you can analyze an unknown solution and determine the presence or absence of Ag^+ , Hg_2^{2+} , and Pb^{2+} .

PROCEDURE

- Prepare a boiling water bath so it will be ready when needed in step b. Prepare precipitates of AgCl , Hg_2Cl_2 , and PbCl_2 in separate labeled test tubes by adding about 5 drops of 6 *M* hydrochloric acid, HCl , to about 1 ml of each of the test solutions: AgNO_3 , $\text{Hg}_2(\text{NO}_3)_2$, and $\text{Pb}(\text{NO}_3)_2$. You may have to cool the test tube containing Pb^{2+} ions to obtain a precipitate. Make a record of your observations for each test.
- Allow the chloride precipitates to settle or use a centrifuge if available. Decant and discard the solution. Add about 2 ml of distilled water to each precipitate. With frequent shaking to stir the precipitate, heat the mixture in a boiling water bath for several minutes. Determine which of the three chlorides is soluble in hot water.
- Remove about 5 drops from the solution containing the most soluble chloride. Use a medicine dropper to transfer the drops to a clean test tube. Add about 5 drops of 0.1 *M* potassium chromate, K_2CrO_4 , solution to the drops of soluble chloride solution.
- Remove the three test tubes from the hot water bath and cool them in cold water.

Shake the test tubes occasionally as you cool them. Allow the precipitates to settle and then decant. Discard the liquid but retain the precipitates for additional tests.

- Add about 3 ml of 6 *M* ammonia water, $\text{NH}_3(\text{aq})$, to each of the precipitates. Shake the test tubes. Add about 3 ml of 6 *M* nitric acid, HNO_3 , to the test tubes in which the precipitate dissolved with ammonia water.
- Examine your record and develop a procedure by which you can identify the presence of one or two of these cations in an unknown solution. Organize your procedure in such a way that another student would be able to follow it.
- Obtain an unknown from your teacher and analyze it for the presence of one or more of the cations tested in this experiment. Report your results together with supporting evidence to your teacher.

OPTIONAL EXERCISES

- Write equations for all reactions occurring in this experiment.
- Construct a flow diagram which summarizes the steps you would use to analyze an unknown containing all three cations.

Experiment 23

Analyzing for Anions

In Experiment 19 you considered the principles involved in developing a scheme of qualitative analysis using a group of unlabeled solutions. In this experiment you will observe and record some characteristic chemical reactions of several negatively charged ions: SO_4^{2-} , CO_3^{2-} , Cl^- , and I^- , using selected reagents. From a study of these observations you will be able to develop a method for identifying each of these anions in the presence of the other ions.

Your work will be typical of the work done by chemists who have developed elaborate schemes which can be used to identify dozens of cations and anions in a given sample. Organize a data table in which you can record your observations for the results obtained by adding each of the reagents to each of the solutions of anions to be studied.

PROCEDURE

- a. The tests can easily be made by putting a few drops of a solution on a plastic film and adding to this a few drops of one of the reagents. DO NOT use the tip of a dropper from a stock bottle for stirring. Doing so could contaminate the solution contained in the bottle. Use a thin glass rod.

Solutions	Anion obtained
Na_2SO_4 , sodium sulfate, 0.1 <i>M</i>	SO_4^{2-}
Na_2CO_3 , sodium carbonate, 0.5 <i>M</i>	CO_3^{2-}
NaCl , sodium chloride, 0.1 <i>M</i>	Cl^-
NaI , sodium iodide, 0.1 <i>M</i>	I^-

- | | Reagents | Cation obtained |
|--|---|------------------|
| | $\text{Ba}(\text{NO}_3)_2$, barium nitrate, 0.1 <i>M</i> | Ba^{2+} |
| | AgNO_3 , silver nitrate, 0.1 <i>M</i> | Ag^+ |
| | HNO_3 , nitric acid, 1.0 <i>M</i> | H^+ |
- b. Test 2–3 drops of each anion solution separately with 1–2 drops of the 0.1 *M* $\text{Ba}(\text{NO}_3)_2$ reagent. Record the results observed. To each mixture that contains a precipitate add 2–3 drops of 1.0 *M* HNO_3 and record any changes you observe.
- c. Test 2–3 drops of each anion solution separately with 1–2 drops of the 0.1 *M* AgNO_3 reagent. Record the results observed. To each mixture that contains a precipitate add

- 2–3 drops of 1.0 *M* HNO₃ and record any changes you observe.
- d. Prepare new samples of the silver precipitates which formed in step c. Add 2–3 drops of 6 *M* aqueous ammonia, NH₃(aq), to the precipitates and record any changes you observe.
- e. Test 2–3 drops of each anion solution separately with 1–2 drops of 1.0 *M* HNO₃ and record the results.
- f. Examine the pattern of reactions shown by your table of observations. Develop a scheme by which you could identify either one or a combination of two of the anions in a single solution. Organize your scheme in such a way that another student could follow it. You may wish to try it out by making up your own “trial unknowns” containing various combinations of the anions.
- g. Obtain an unknown from your teacher and analyze it for the four anions. Report your results. Include supporting evidence for your conclusions.

PROCESSING THE DATA

- 1) Write net ionic equations for the precipitation reactions which occurred when solutions of the anions were mixed with the reagent containing Ba²⁺ ions.
- 2) Do the same for the precipitation reactions which occurred in step c with the Ag⁺ ions.
- 3) Write equations for the reactions which occurred when the precipitates were acidified with 1.0 *M* HNO₃.
- 4) Write equations for the reactions which occurred when the silver precipitate reacted with aqueous ammonia to form the complex ion, Ag(NH₃)₂⁺.



Experiment 24

A Study of Chemical Reactions

What is a chemical reaction?

It is a rearrangement of atoms in which bonds are broken and/or formed. But we cannot observe this sort of change directly. What clues should we look for when deciding whether a chemical reaction has occurred? The following list is not exhaustive, but does discuss some of the easier-to-observe clues.

- i. *A precipitate is formed.* If a precipitate forms, new strong bonds must have formed, which cannot be broken by reactions with the other chemicals available.
- ii. *A gas forms.* Again this must mean new bonds form and/or old bonds break to make a substance not previously present.
- iii. *A color change.* Most color is the result of certain energy changes electrons experience. The possible changes are determined by the environment of the electrons. Changes in bonding cause changes in environment.
- iv. *Heat absorbed or evolved.* Bond breaking requires energy, and bond forming releases energy. Often these are not equal and the overall reaction is endothermic or exothermic.

In this experiment you will also have an opportunity to observe the rate of chemical reactions. Some reactions are fast, others slow. What affects rate? You will test temperature, concentration, nature of reactants, and catalysts.

Refer to Laboratory Instructions on page ix, and especially to items 5, 6, and 7 regarding the precautions to be observed in handling, tasting, and smelling chemicals.

For each step in the Procedure, record a description of the reactants, observations made when potential reactants were mixed, and reasons for believing a chemical reaction has or has not taken place. Note that volumes are given to only one significant figure. You should be able to estimate them without using a graduated cylinder. Unless you are told to save a solution, it will not be needed in future steps.

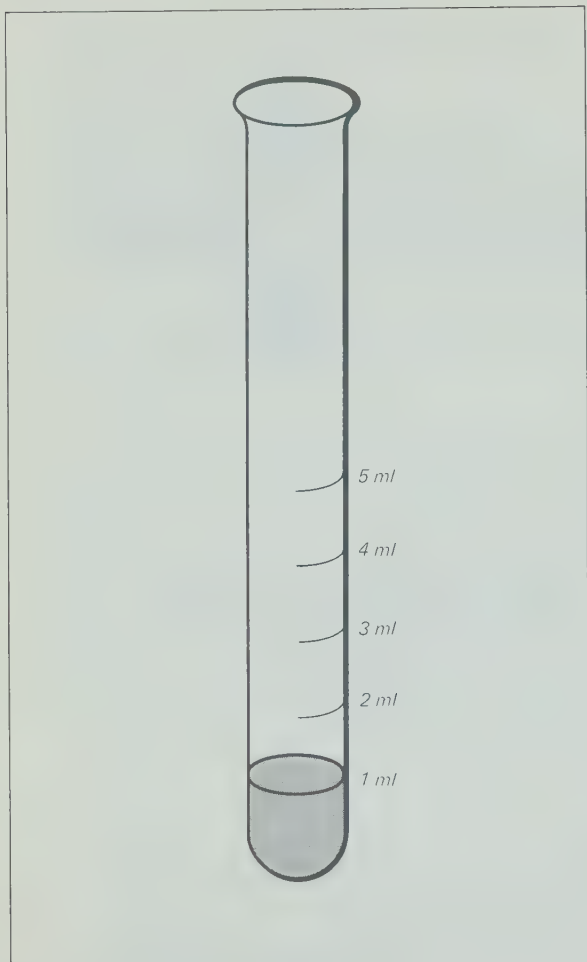


Figure 24-1

A full-scale drawing of a calibrated 13 × 100 mm test tube.

PROCEDURE

Part I

- a. To 5 ml of tap water in a test tube add 15 drops, a drop at a time, of 18 *M* sulfuric acid, H_2SO_4 . **Always add concentrated acid to water; never add water to concentrated acid.** Record your observations. Save the diluted acid (about 2 *M*) to use in Part II f.

- b. To 5 ml of tap water in a test tube add 3 small pellets of solid sodium hydroxide, NaOH(s) . *Do not handle sodium hydroxide pellets with your fingers.* Cover the test tube and shake it gently. Record your observations. Save the solution for Part II e.
- c. To 1 ml of solid ammonium chloride, $\text{NH}_4\text{Cl(s)}$, in a test tube add 5 ml of tap water. Cover the tube and shake it gently. Record your observations.
- d. Repeat step c, except use 1 ml of solid sodium acetate, $\text{NaCH}_3\text{COO(s)}$ in place of the $\text{NH}_4\text{Cl(s)}$.

Part II

- e. Add 1 ml of the sodium hydroxide solution prepared in Part Ib to 5 ml of tap water. Add a few drops of a solution of phenolphthalein (an indicator solution). Cover the tube and shake it gently.
- f. Repeat the test of step e except use 1 ml of the sulfuric acid solution prepared in Part Ia in place of the sodium hydroxide solution.
- g. Place a small amount (about $\frac{1}{4}$ ml) of solid sodium sulfite, $\text{Na}_2\text{SO}_3\text{(s)}$, in a test tube. Add cautiously about 3 ml of 6 *M* hydrochloric acid, HCl .
- h. Place 5 ml of 0.1 *M* acidified ferrous sulfate, FeSO_4 , in a test tube. Add 10 drops of 0.1 *M* potassium permanganate, KMnO_4 , one drop at a time, shaking the test tube after the addition of each drop.
- i. To 1 ml of 0.1 *M* sodium chloride, NaCl , in a test tube, add 1 ml of 0.1 *M* potassium bromide, KBr .
- j. Place about $\frac{1}{2}$ ml of powdered lead dioxide, PbO_2 , in a test tube. Heat over a burner flame and note any changes. Light a wooden splint, blow out the flame, and quickly thrust it into the test tube while the splint is still glowing. How do you account for the result? Set the test tube aside to cool. Do not attempt to wash this tube.

Part III

- k. Put 20 ml of 0.1 *M* sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$, in a beaker. Add 1 ml (20 drops) of 2 *M* H_2SO_4 . Swirl to mix. To each of four test tubes add 5 ml of the above solution.

(1) Place two of the test tubes in a hot water bath (40–50°C) so that both will be at the same temperature. To one of these tubes add 5 drops of 0.1 *M* manganous sulfate, MnSO_4 . Next add 2 drops of 0.1 *M* KMnO_4 to each of the two test tubes. Shake each tube to mix, and note the time of reaction for each to reach the same end products.

(2) To *one* of the other two test tubes, at room temperature, add 5 drops of 0.1 *M* manganous sulfate, MnSO_4 . Then to each of these two test tubes add 2 drops of 0.1 *M* KMnO_4 . Shake each tube to mix, and compare the time of reaction for each as in (1) above.

- l. Arrange five test tubes in order in a test tube rack. Add to each tube as indicated below.

Tube Number	Add 5 ml of
1	6 <i>M</i> hydrochloric acid, HCl
2	6 <i>M</i> acetic acid, CH_3COOH
3	1 <i>M</i> HCl
4	1 <i>M</i> CH_3COOH
5	0.1 <i>M</i> HCl

To each test tube add a small chip of calcium carbonate, $\text{CaCO}_3(\text{s})$. Record the relative rates of reaction observed.

Part IV

- m. Place 1 ml of 0.1 *M* sodium chloride, NaCl , in a test tube and 1 ml of 0.1 *M* potassium chromate, K_2CrO_4 , in another tube. Add a few

drops of 0.2 *M* silver nitrate, AgNO_3 , to each. Note the results.

- n. To the tube containing K_2CrO_4 and AgNO_3 , add 0.1 *M* NaCl solution dropwise. Shake the test tube after each drop. Continue adding NaCl solution until a precipitate changes color.
- o. Mix in a test tube 1 ml of 0.1 *M* sodium chloride, NaCl , and 1 ml of 0.1 *M* potassium chromate, K_2CrO_4 . Add 0.2 *M* silver nitrate, AgNO_3 , one drop at a time, shaking the tube after the addition of each drop. Continue to add the AgNO_3 solution until no further change is observed.

PROCESSING THE DATA

- 1) In which of the experiments was there no evidence of a chemical reaction?
- 2) Which chemical reactions produced a new phase?
- 3) Which reactions in this experiment were exothermic? Which were endothermic?
- 4) In which reactions did an increase in temperature affect the rate?
- 5) In which reactions did an increase in concentration affect the rate?
- 6) In Part IIIk what effect on the rate of the reaction did adding the MnSO_4 solution have?
- 7) What evidence did you observe to indicate that in some of the reactions part of the reactants was not used up?

A QUESTION TO WONDER ABOUT

Account for the results obtained when solutions of hydrochloric acid and of acetic acid — of the same concentration — reacted with $\text{CaCO}_3(\text{s})$.



Experiment 25

Energy of Combustion

Everyone knows the flame of a candle releases energy. In this experiment, you will determine *how much* heat is released.

What is the source of this energy? We find that energy is needed to break chemical bonds and energy is released when chemical bonds form. The main source of energy for the candle flame is found in the balance of these energy exchanges. When a candle burns, more energy is released when the bonds form to make the products than is needed to break the bonds in the reactants. The reactants are mainly candle wax and oxygen. The products are carbon dioxide, water, and soot.

The amount of heat produced will depend on the amount of wax burned. If results from different experiments are to be compared, the same amount of wax will have to be burned in each experiment. This is very difficult to do experimentally, but very simple with a little arithmetic. If the number of calories of heat produced is divided by the grams of wax burned, the result will be calories of heat produced per one gram of wax burned.

The procedure is to determine the temperature increase of a known mass of water by adding to it the heat produced by burning a known mass of candle wax.

Before the experiment prepare a data sheet on which you can enter the data collected in the following procedure.

PROCEDURE

- a. Attach a candle to a tin can lid.
- b. Set up the apparatus as shown in Figure 25-1. First, without the large can in place, adjust the height of the small can so that the bottom of the can is about 2 inches above the tip of the wick. The tip of the flame should almost but not quite reach the bottom of the can.
- c. Weigh the candle and lid to the nearest 0.01 gram. Record the mass of candle and lid in your data table. Note the balance number so that when you weigh the candle again you can use the same balance. Place

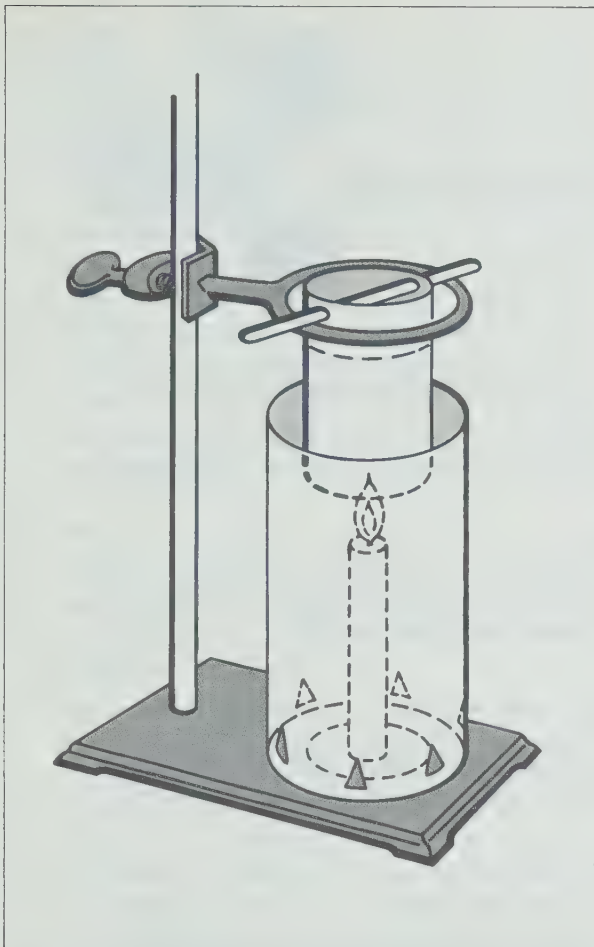


Figure 25-1

Apparatus for determining the heat of combustion.

- the candle and lid in position inside the large can which protects the candle from drafts.
- Fill the small can about two-thirds full with cold tap water. Do not measure the volume of the water at this time.
 - Cool the water with ice, if necessary, so that its temperature is about $10\text{--}15^\circ\text{C}$ below room temperature. Add the ice directly to the water. Remove any remaining ice when the desired temperature has been reached.
 - Read and record the temperature of the water to the nearest 0.1°C . Light the candle

and quickly place the can of water in position. Heat the water, stirring it gently, until it reaches a temperature about as much above room temperature as it was below at the start. Carefully blow out the candle flame. Continue to stir the water, while watching the thermometer reading, until the highest temperature is reached. Record the highest temperature to the nearest 0.1°C .

- Weigh the candle on the same balance that was used to weigh it before. Make certain that any drippings from the candle are weighed with it. Record the mass.
- Measure the volume (± 1 ml) of water heated.
- Repeat the experiment if time permits.

PROCESSING THE DATA

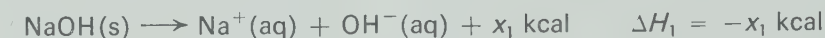
- Determine the mass of candle wax burned.
- Determine the mass of water heated. One ml of water has a mass of one gram.
- Determine the temperature change, Δt , of the water.
- Calculate the quantity of heat absorbed by the water in the can.
- Calculate the heat of combustion of candle wax (calories per gram).
- Do you think an experiment using a more refined calorimeter would give a higher or a lower value than the one you determined in Question 5? Explain.
- Most candles are made of a mixture of waxes. Assume yours was a pure wax with the formula $\text{C}_{25}\text{H}_{52}$. Calculate the heat of combustion of $\text{C}_{25}\text{H}_{52}$, $\Delta H_{\text{combustion}}$, in kcal/mole.
- (Optional) Assume some black soot formed on the bottom of the can of water during your experiment. Would this contribute to a high or a low value for $\Delta H_{\text{combustion}}$? Explain.

Experiment 26

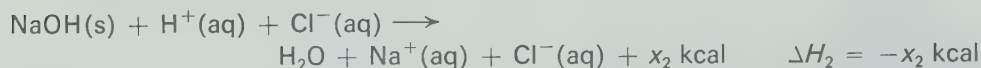
Heat of Reaction

In this experiment you will measure and compare the quantity of heat energy released in three exothermic chemical reactions.

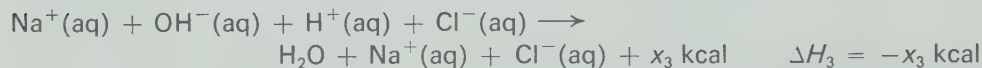
Reaction 1. Solid sodium hydroxide dissolves in water to form an aqueous solution of ions.



Reaction 2. Solid sodium hydroxide reacts with an aqueous solution of hydrogen chloride to form water and an aqueous solution of sodium chloride.



Reaction 3. An aqueous solution of sodium hydroxide reacts with an aqueous solution of hydrogen chloride to form water and an aqueous solution of sodium chloride.



You will use a Styrofoam cup as a calorimeter. Assume that the heat of reaction is used to change the temperature of the aqueous solution only and that the amount used to warm the cup or the surrounding air is negligible. Further, you may assume that the energy required to change the temperature of one milliliter of solution is the same (within the limits of experimental uncertainty) as that required to change the temperature of one gram of water through the same change in temperature.

PROCEDURE

Reaction 1

- Using water at about 1 or 2 degrees below room temperature, measure 200 ± 1 ml of it into a Styrofoam cup.
- Obtain about 2 grams of solid sodium hydroxide pellets, NaOH(s) , and record its mass to the nearest 0.01 gram. Since sodium hydroxide picks up moisture from the air, it

is necessary to weigh it and proceed to the next step without delay.

- When you are ready to add the measured amount of sodium hydroxide to the water in the calorimeter, record the starting temperature of the water to the nearest 0.1°C . Pour the pellets of sodium hydroxide into the water and stir the solution gently until all of the solid has dissolved. Record the highest temperature reached during the reaction.

Dispose of the solution as directed by your teacher. Rinse the cup and the thermometer thoroughly before proceeding to *Reaction 2*.

Reaction 2

- d. The procedure for *Reaction 2* is the same as for *Reaction 1* except that 200 ± 1 ml of 0.25 *M* hydrochloric acid solution is used in place of the water. After measuring 200 ± 1 ml of the HCl solution into the Styrofoam cup, proceed as before with steps b and c of the procedure.
- e. Discard the solution and rinse the cup before proceeding with *Reaction 3*.

Reaction 3

- f. Measure 100 ± 1 ml of 0.50 *M* hydrochloric acid solution into a Styrofoam cup. Obtain an equal volume of 0.50 *M* sodium hydroxide solution in a clean beaker.
- g. Record the temperature of each solution to the nearest 0.1°C . Use the same thermometer for both solutions, but be sure to rinse it thoroughly before transferring it from one solution to another. Pour the sodium hydroxide solution into the hydrochloric acid solution in the Styrofoam cup. Stir the solution gently, and record the highest temperature obtained during the reaction. Discard the solution.

PROCESSING THE DATA

- 1) Prepare a table such as the one shown at the bottom of this page and make the necessary calculations in order to complete it.
- 2) Use the ΔH notation to express the energy released per mole of NaOH for each reaction: ΔH_1 , ΔH_2 , and ΔH_3 . Use two significant figures in all calculations.
- 3) Write net ionic equations for *Reactions 2* and *3*.
- 4) ΔH_1 , in *Reaction 1*, represents the energy of solution for one mole of NaOH(s). Look at the net ionic equations for *Reactions 2* and *3*, and make a similar statement concerning the significance of ΔH_2 and ΔH_3 .
- 5) Compare ΔH_2 with $(\Delta H_1 + \Delta H_3)$. Account for any similarity or difference.
- 6) Calculate the percent difference between ΔH_2 and the sum $(\Delta H_1 + \Delta H_3)$. Assume ΔH_2 to be correct.
- 7) Suppose you had used 4.00 grams of NaOH(s) in *Reaction 1*. What would have been the number of calories released in the reaction? What effect would this have on your calculation of ΔH_1 ?

Reaction	Δt	Heat Energy Released	Moles of NaOH Used	Energy Released per mole NaOH
1				
2				
3				

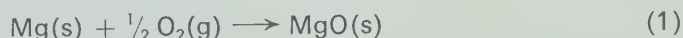


Experiment 27

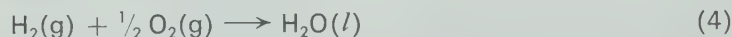
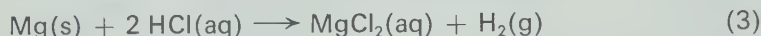
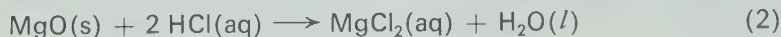
Heat of Reaction for the Combustion of Magnesium

When a reaction can be expressed as the algebraic sum of a sequence of two or more simpler reactions, the heat of reaction is the algebraic sum of the heats of these simpler reactions. This generalization has been found to be true for every reaction that has been tested.

In this experiment you will use this generalization to determine the ΔH for a reaction that is difficult to measure directly using a Styrofoam calorimeter. Magnesium metal burns very rapidly, as you have observed in photo flashbulbs, releasing much light and heat. The reaction is represented by the equation



This equation can be obtained by combining equations (2), (3), and (4).



Combine these three equations to obtain equation (1) before doing the experiment. See item 1 under *Processing the Data*.

Heats of reaction for equations (2) and (3) can easily be determined. The ΔH for reaction (4) can be obtained from a table of values for previously measured reactions, page 198 in your Textbook.

PROCEDURE

For both of these reactions, measure the HCl solution into a Styrofoam calorimeter. Record the temperature of the solution and add either MgO or Mg to the solution. Stir the mixture and record the highest temperature reached during each reaction. Measure temperature to the nearest 0.1°C and mass to the nearest centigram.

Reaction 2

Measure the change in temperature that occurs as about 1 gram of magnesium oxide, MgO, is added to 100 ± 1 ml of 1.00 M HCl solution.

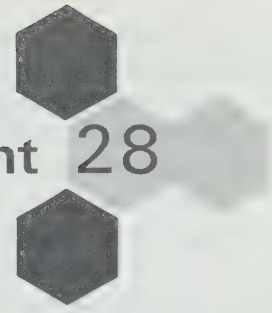
Reaction 3

Measure the change in temperature that occurs as about 0.5 gram of magnesium

metal, Mg, is reacted with 100 ± 1 ml of 1.00 M HCl solution.

PROCESSING THE DATA

- 1) Combine equations (2), (3), and (4) to obtain equation (1).
- 2) Calculate the change in temperature, Δt , for each reaction, (2) and (3).
- 3) Calculate the kcal of energy released for each reaction. Assume that one calorie of energy is required to change the temperature of one ml of solution one Celsius degree.
- 4) Calculate the kcal/mole of Mg and kcal/mole MgO.
- 5) Determine the ΔH for reaction (1). Report ΔH in kcal/mole of MgO.



Experiment 28

A Study of Reaction Rates

Perhaps you have noticed that a fire burns faster when fanned by a gentle breeze, and that a candle burns more brightly and vigorously when burning in pure oxygen rather than in air. Or perhaps you have observed how poorly a fire burns if not enough air is supplied to it. Other chemical reactions are similarly affected by changes in concentration of one or more of the reactants. Chemical reactions are also affected by changes in temperature. Just how concentration and temperature affect chemical reactions are questions to be investigated in this experiment.

In Part I you will vary the concentration of the reacting species. In Part II you will vary the temperature. In Part III you will study the effect of adding a catalyst. In order to study the effect of one variable you will keep other variables constant.

The reaction you will study involves three different solutions as described below:

Solution A. 0.20 *M* potassium iodide, KI, a source of I^- . This solution also includes some starch which acts as an indicator for iodine.

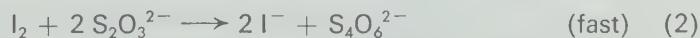
Solution B. 0.0050 *M* sodium thiosulfate, $Na_2S_2O_3$, a source of $S_2O_3^{2-}$.

Solution C. 0.10 *M* ammonium peroxydisulfate, $(NH_4)_2S_2O_8$, a source of $S_2O_8^{2-}$.

We are primarily interested in the reaction between iodide ions, I^- , and peroxydisulfate ions, $S_2O_8^{2-}$, as represented in equation (1).

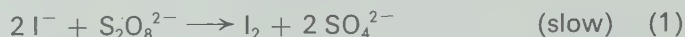


In order to study the rate of a reaction we need to be able to determine how fast one of the products is formed or how fast one of the reactants is consumed. In this reaction we can determine how long it takes to form a certain amount of I_2 . In order to do this we make use of the very fast reaction



You will notice that in reaction (1) iodine is formed. But in reaction (2) thiosulfate ions, $S_2O_3^{2-}$, react with iodine to change it to iodide, I^- . Since reaction (2) is very fast, the iodine produced in reaction (1) is used up about as fast as it is produced. As soon as all of the thiosulfate ions have reacted, the iodine produced in reaction (1) remains as I_2 , and as such reacts with starch (present in *Solution A*) to form a blue colored substance. Only a very small amount of iodine is required to form the blue colored substance with starch.

In this first set of experiments, the iodide ion concentration will be varied for three of the mixtures. The peroxydisulfate ion concentration will be varied for three other mixtures. Another mixture, number 1, is used as a point of reference. The amount of thiosulfate ion is kept constant for all seven mixtures. This fixed amount of thiosulfate ion will convert the same amount of iodine to iodide for each of the trials. As soon as the thiosulfate is used up, iodine will be available to react with starch and a blue color will appear. By noting the time required for the blue color to appear, we can determine the effect of iodide concentration and peroxydisulfate concentration on the rate of the reaction



You will be measuring the different times required to produce one certain amount of iodine. The cause of any observed time variation will be due to changes in the iodide ion or peroxydisulfate ion concentrations.

PROCEDURE

Part I. The Effect of Concentration

There are seven different combinations of *Solutions A, B, and C* to be investigated in this part of the experiment. They are listed in Table 28-1. Working in pairs, you should investigate the reaction using *Mixture 1* and any others assigned to you by your teacher. Data for each of the mixtures will be averaged and shared by all members of the class. The solutions for this part of the experiment should all be at room temperature.

- a. Use clean graduated cylinders marked A, B, and C. Carefully measure the volumes of the three solutions for each mixture as indicated in Table 28-1.

- b. Pour 10.0 ml of *Solution B* into a 100 ml beaker. Place a thermometer into the beaker. One partner should be prepared to give a signal when to pour the solutions together and record the time of pouring. Upon receiving a signal, pour both solutions, *A* and *C*, quickly and simultaneously into the beaker. Stir the solution with a glass stirring rod to mix it thoroughly. Record the temperature of the mixture. Watch for the first sign of blue to appear and record the time at that instant.
- c. Repeat the experiment using *Mixture 1* until you can duplicate the time of reaction within 2 or 3 seconds. Then use any other mixture assigned by your teacher. Obtain averages of class data for all the mixtures.

TABLE 28-1

(All Volumes in ml)

Mixture	Solution A, source of I^-	Solution B, source of $\text{S}_2\text{O}_8^{2-}$	Solution C, source of $\text{S}_2\text{O}_8^{2-}$
1	20.0	10.0	20.0
2	15.0 + 5.0 water	10.0	20.0
3	10.0 + 10.0 water	10.0	20.0
4	5.0 + 15.0 water	10.0	20.0
5	20.0	10.0	15.0 + 5.0 water
6	20.0	10.0	10.0 + 10.0 water
7	20.0	10.0	5.0 + 15.0 water

Part II. The Effect of Temperature

There are five different combinations of *Solutions A, B, and C* at different temperatures to be investigated in this part. See Table 28-2. Notice that all the mixtures use the same amount of each solution. You will be assigned one or more of the mixtures for certain temperature ranges. You should record the temperature observed when the solutions are first mixed. This temperature should be within the range assigned to you. Data for the other mixtures can be obtained by averaging class data.

- d. Measure the solutions into separate, labeled test tubes. Warm or cool the solutions to the desired temperature by placing the test tubes

into a water bath. Use ice to cool the water bath below room temperature, but do not dilute the solutions with melted ice. Place a thermometer in the tube containing *Solution B* only. Assume *Solutions A* and *C* have the same temperature as *B*.

- e. After the desired temperature has been reached, pour *Solution B* into a 100 ml beaker. Place the thermometer in *Solution B*. Pour the other two solutions simultaneously into *Solution B* and note the time. Stir the mixture with a glass stirring rod and watch for the first sign of blue color. Record the time when the first sign of blue color appears.

Table 28-2
(All Volumes in ml)

Mixture	Solution A, source of I^-	Solution B, source of $S_2O_3^{2-}$	Solution C, source of $S_2O_8^{2-}$	Temperature Range, °C
8	20.0	10.0	20.0	5–10
9	20.0	10.0	20.0	10–20
10	20.0	10.0	20.0	20–30
11	20.0	10.0	20.0	30–40
12	20.0	10.0	20.0	40–45

Part III. The Effect of a Catalyst

- f. Repeat the experiment as in Part II, but this time add 4 drops of 0.01 *M* copper sulfate solution to each 20.0 ml of *Solution C* used. Use the mixtures as listed in Table 28-3 for the temperature ranges assigned by your teacher.

TABLE 28-3

Each mixture requires

20.0 ml *Solution A* (source of I^-)

10.0 ml *Solution B* (source of $S_2O_3^{2-}$)

20.0 ml *Solution C* (source of $S_2O_8^{2-}$)
with 4 drops of 0.01 *M* copper sulfate
solution added.

Mixture	Temperature Range, °C
13	5–10
14	10–20
15	20–30
16	30–40
17	40–45

PROCESSING THE DATA

Part I. The Effect of Concentration

Mixture	ml of Solution A, 0.20 M KI	Moles of I^-	ml of Solution C, 0.10 M $(NH_4)_2S_2O_8$	Moles of $S_2O_8^{2-}$	Elapsed Time (sec)
1	20.0		20.0		
2	15.0		20.0		
3	10.0		20.0		
4	5.0		20.0		
5	20.0		15.0		
6	20.0		10.0		
7	20.0		5.0		

- 1) Prepare a table such as the one shown above. Make the calculations needed to complete it.
- 2) In each of the mixtures you used 10.0 ml of *Solution B* (0.0050 M $Na_2S_2O_3$). Calculate, using equation (2), how many moles of I_2 will react with this much thiosulfate ion, $S_2O_3^{2-}$. The elapsed time for each reaction is a measure of the time it takes to use up this much $S_2O_3^{2-}$ and to produce n moles of I_2 according to equation (1).
- 3) Make a graph by plotting moles of iodide ion (*Solution A*) vs elapsed time for *Mixtures* 1–4. Plot elapsed time for reactions along the horizontal axis.
- 4) Make another graph, plotting moles of peroxydisulfate ion (*Solution C*) vs elapsed time for *Mixtures* 1, 5, 6, and 7. Plot elapsed time for reactions along the horizontal axis.
- 5) How does changing the concentration of reactants affect the elapsed time for the reaction?
- 6) How many moles of $S_2O_8^{2-}$ are not used if all of the I^- in *Mixture* 3 is completely changed to I_2 ?

Part II. The Effect of Temperature

- 7) Prepare a table such as shown and make

the calculations necessary in order to complete the table.

Mixture	Temperature (°C)	Elapsed Time (sec)
8		
9		
10		
11		
12		

- 8) Make a graph plotting temperature of the mixture vs elapsed time for *Mixtures* 8 through 12. Plot time along the horizontal axis.
- 9) What generalization can you deduce from these data?
- 10) How does a temperature change of about 10C° affect the elapsed time for the reaction?

Part III. The Effect of a Catalyst

- 11) Prepare a graph plotting temperature of the mixture vs elapsed time, on the same graph prepared in Part II. Use the time-temperature data for *Mixtures* 13–17. Label this curve "catalyzed" and the other (from Part II) "uncatalyzed."
- 12) What generalization can you deduce from a comparison of the two curves?



Experiment 29

Chemical Equilibrium

In this experiment you will study in a quantitative manner the reaction $\text{Fe}^{3+}(\text{aq}) + \text{SCN}^{-}(\text{aq}) \rightleftharpoons \text{FeSCN}^{2+}(\text{aq})$ which was presented qualitatively as a class experiment by your teacher. This time you will determine the concentration of each of the ions at equilibrium, and then seek an expression that relates these quantities mathematically in a simple, convenient manner.

Some of the concentrations will be determined colorimetrically. If you have ever looked critically at a full glass of a colored liquid, such as iced tea, you know that the color intensity as viewed through the side of the glass is much less than the color intensity as viewed from the top down. The color of the light depends on the number of colored species the light interacts with as it travels to your eye. This depends upon the concentration of the colored substance and on the depth of the solution. Thus, a 1 cm depth of a 1 *M* colored solution will appear to have the same color intensity as a 2 cm depth of a 0.5 *M* solution of the same material.

By expressing this relationship mathematically, we get an equation which allows us to calculate an unknown concentration. We must know one concentration and the heights of the two solutions required to produce the same color. The equation is $C_1 h_1 = C_2 h_2$ where C_1 equals a known concentration of colored species in tube 1 and C_2 equals an unknown concentration of colored species in tube 2; h_1 and h_2 are the heights of the solutions in tubes 1 and 2 that produced the same intensity of color when viewed through distance h_1 or h_2 .

In preparing the standard solution in step a of this experiment, you will use a low, known concentration of thiocyanate ion, $\text{SCN}^{-}(\text{aq})$, and add a large excess of ferric ion, $\text{Fe}^{3+}(\text{aq})$. You can assume that essentially all of the thiocyanate ion will be used in forming the colored complex thiocyanoferric(III) ion, $\text{FeSCN}^{2+}(\text{aq})$, and that the equilibrium concentration of the $\text{FeSCN}^{2+}(\text{aq})$ ion will be essentially the same as the concentration of the $\text{SCN}^{-}(\text{aq})$ ion with which you started. Thus we know C_1 , the equilibrium concentration of the colored species FeSCN^{2+} .

Prepare a data sheet for recording the height of solution in each of four different tubes and the height of solution in the standard tube required to produce matching color intensities.

PROCEDURE

- a. Line up five clean test tubes, all of the same diameter, and label them 1, 2, 3, 4, 5. Add 5.0 ml of 0.00200 *M* potassium thiocyanate, KSCN, to each of these five test tubes.
- b. To test tube 1 add 5.0 ml of 0.200 *M* ferric nitrate, $\text{Fe}(\text{NO}_3)_3$. This tube will be used as the standard.
- c. Measure 10.0 ml of 0.200 *M* $\text{Fe}(\text{NO}_3)_3$ in your graduated cylinder and fill to the 25.0 ml mark with distilled water. Pour the solution into a clean dry beaker. Stir to mix thoroughly. Measure 5.0 ml of this solution and pour it into test tube 2. Save the remainder of the $\text{Fe}(\text{NO}_3)_3$ solution for Part d.
- d. Pour 10.0 ml of the solution from the beaker into your graduated cylinder. Discard the remainder. Finish filling the graduated cylinder to the 25.0 ml mark with distilled water, and pour the solution into a clean dry beaker to mix. Pour 5.0 ml of this solution into test tube 3.
- e. Continue diluting the solution in this manner
- f. Now the problem is to compare the solutions in each of the test tubes separately with the standard tube (number 1). Start by removing solution from tube 1 until the color matches that in tube 2. Remove solution from tube 1 with a clean dry dropper, and store it in a clean dry beaker. Matching colors is difficult under the best of conditions. As an aid, wrap the tubes separately in white paper. This will exclude extraneous side light. Look down through the tubes while they are pointed toward a uniform white light. See Figure 29-1. It sometimes helps to remove an excess of liquid from tube 1 and return some dropwise until a color match is achieved. When the color intensities appear the same, measure the depth of each solution to the nearest millimeter and record this.
- g. Repeat this color comparing procedure with test tubes 1 and 3, 1 and 4, and finally 1 and 5.

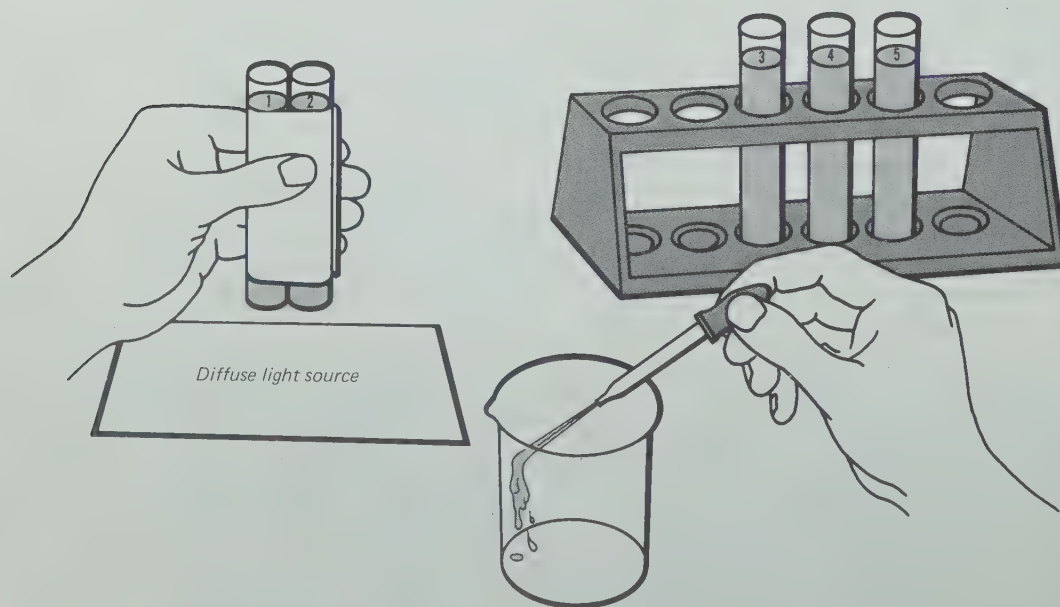


Figure 29-1 Comparing the color of two samples over a diffuse light source.

PROCESSING THE DATA

- 1) Carefully follow through the process of diluting the original 0.200 M $\text{Fe}(\text{NO}_3)_3$ solution to determine the initial concentration in moles of Fe^{3+} per liter, $[\text{Fe}^{3+}]_{\text{initial}}$, in each tube. This is the concentration before any Fe^{3+} has had a chance to react. Carry two significant figures through all calculations.
- 2) The solution in each tube was made by diluting 5.0 ml of 0.00200 M KSCN with 5.0 ml of a $\text{Fe}(\text{NO}_3)_3$ solution. Calculate $[\text{SCN}^-]_{\text{initial}}$.
- 3) To calculate the equilibrium concentration of the colored species, $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$, recall that in tube 1 we assume all SCN^- was used to form FeSCN^{2+} . To determine $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$ in the other tubes, use the relation $C_n = C_1 \frac{h_1}{h_n}$ where n refers to tubes 2, 3, 4, 5.
- 4) Determine $[\text{Fe}^{3+}]_{\text{equilibrium}}$ for each tube. This is $[\text{Fe}^{3+}]_{\text{initial}}$ reduced by the concentration needed to produce $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$.
- 5) For each tube determine $[\text{SCN}^-]_{\text{equilibrium}}$. This is $[\text{SCN}^-]_{\text{initial}}$ reduced by the amount needed to produce $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$.
- 6) The problem now is to see if there is a regularity among these equilibrium concentrations. Since regularities are often given

a mathematical form, we will seek one here. Although there are many ways these equilibrium concentrations can be mathematically manipulated, we will try only three.

For each tube, calculate the values of the following three ratios, using the equilibrium concentration of each ion.

- a) $\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^-]}$
- b) $\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^-]}$
- c) $\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$

Use two significant figures in all calculations.

- 7) For each of the combinations a, b, c, in Question 6, calculate the value of the ratio $\frac{\text{largest numerical value for tubes 2-5}}{\text{smallest numerical value for tubes 2-5}}$
- 8) For the combination giving the smallest value for the ratio in Question 7, calculate the value of the ratio $\frac{\text{largest concentration}}{\text{smallest concentration}}$ for each of the ions used.
- 9) Restate in words the combination (A, B, or C) giving the smallest value of the ratio in Question 7.



Experiment 30

Applying Le Chatelier's Principle

Most of the chemical reactions you have observed in the laboratory seemed to go to completion — that is, the reactants appeared to be used up in forming the products. The products did not seem to change back and form the reactants. The reaction seemed to go only in one direction.

In this experiment you will study some reactions where appreciable reversibility is found. Reactants will form products, and products will form reactants. A change in the relative amounts of reactants and products can be readily observed by noting color changes or the formation of a precipitate. In an aqueous solution the chromate ion, CrO_4^{2-} , can be converted to the dichromate ion, $\text{Cr}_2\text{O}_7^{2-}$, and conversely, the $\text{Cr}_2\text{O}_7^{2-}$ ion can be converted to the CrO_4^{2-} ion. The extent to which these reactions take place depends upon the concentration of the hydrogen ion in the solution. The H^+ concentration can be increased by adding a source of H^+ — hydrochloric acid, HCl . The H^+ concentration can be decreased by adding a solution of sodium hydroxide, a source of hydroxide ion. The OH^- reacts with H^+ to form H_2O .

PROCEDURE

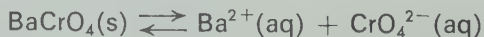
Part I. The Chromate Ion — Dichromate Ion Equilibrium

- Obtain about 5 ml of 0.1 *M* potassium chromate, K_2CrO_4 , and 5 ml of 0.1 *M* potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, in separate test tubes. These solutions will serve as sources for the ions, CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$. Record in your notebook the color of each solution.
- Place 10 drops (about 0.5 ml) of each solution into separate 13 × 100 mm test tubes. Add some dilute sodium hydroxide, 1 *M* NaOH , a drop at a time, alternately to each solution until a color change is noted in one of the tubes. Record the colors now. Retain these tubes for step e.
- Repeat the procedure of step b with fresh solutions in clean test tubes; except add dilute hydrochloric acid, 1 *M* HCl , drop by

drop, alternately to each test tube. Record the color change observed. Retain these test tubes for step d.

- Add 1 *M* NaOH , drop by drop, to either of the tubes produced in step c until a change is noted.
- Add 1 *M* HCl , drop by drop, to either of the tubes produced in step b until a change is noted.

Part II. The Equilibrium of Solid Barium Chromate, $\text{BaCrO}_4(\text{s})$, with a Saturated Solution of Its Ions:



- Place 10 drops (about 0.5 ml) of 0.1 *M* K_2CrO_4 in a clean test tube. Add 2 drops of 1 *M* NaOH . Add 0.1 *M* $\text{Ba}(\text{NO}_3)_2$, barium nitrate, a drop at a time, until a change is noted. Record the result. Retain this test tube for step h.

- g. Place 10 drops of 0.1 *M* $\text{K}_2\text{Cr}_2\text{O}_7$ into a clean test tube. Add 2 drops of 1 *M* HCl , then 10 drops of 0.1 *M* $\text{Ba}(\text{NO}_3)_2$. Record the result. Retain this test tube for step i. Record your conclusions about the relative solubilities of $\text{BaCrO}_4(\text{s})$ and $\text{BaCr}_2\text{O}_7(\text{s})$ from your observations in steps f and g.
- h. To the test tube from step f add 1 *M* HCl , drop by drop, until a change is noted. Record your observation.
- i. To the test tube from step g add 1 *M* NaOH , drop by drop, until a change is noted. Record your observation.
- j. Suggest a way to reverse the changes and reactions you observed in step h. Do the same for step i. Try these experiments.
- k. Place 10 drops of 0.1 *M* $\text{K}_2\text{Cr}_2\text{O}_7$ in one test tube and the same amount of 0.1 *M* K_2CrO_4 in another test tube. Add a few drops of 0.1 *M* $\text{Ba}(\text{NO}_3)_2$ to each. Record your observation, including the relative amounts of product formed.

Part III. Optional. Further Tests on the Chromate-Dichromate Ion Equilibrium

Repeat steps a and b, using the solutions of $\text{K}_2\text{Cr}_2\text{O}_7$ and K_2CrO_4 but testing them separately with several drops of dilute solutions of each of the following substances: CH_3COOH , HNO_3 , $\text{Ca}(\text{OH})_2$, H_2SO_4 , KOH , $\text{C}_2\text{H}_5\text{OH}$, NH_3 . Record any changes in color noted.

PROCESSING THE DATA

Questions for Part I

- 1) What can you conclude about the change $2\text{CrO}_4^{2-} \longrightarrow \text{Cr}_2\text{O}_7^{2-}$ and its dependence on hydrogen ions, H^+ , as noted in steps c and e? Produce a balanced equation by adding the proper number of H^+ ions and H_2O molecules to the appropriate sides of the equation.
- 2) What can you conclude about the reverse change $\text{Cr}_2\text{O}_7^{2-} \longrightarrow 2\text{CrO}_4^{2-}$ and its dependence on hydroxide ions, as noted in steps b and d? Produce a balanced equation by adding the proper number of OH^- ions and H_2O molecules to the appropriate sides of the equation.

Questions for Part II

- 3) From your observation in step k, what can you conclude about the relative equilibrium concentrations of CrO_4^{2-} ion in each of the solutions 0.1 *M* $\text{K}_2\text{Cr}_2\text{O}_7$ and 0.1 *M* K_2CrO_4 ?
- 4) Use the equations you balanced in questions 1 and 2 to explain the results you obtained in steps h, i, and j.
- 5) Make a statement summarizing your results with the chromate ion-dichromate ion equilibrium. Include the application of the Principle of Le Chatelier.

Questions for Part III

- 6) a. Which substances in solution caused the color to change from that of the $\text{Cr}_2\text{O}_7^{2-}$ ion to that of the CrO_4^{2-} ion?
b. Which substances in solution caused the reverse color change?
- 7) What ionic species do the solutions you listed in Question 6a have in common? Answer the same question for the solutions listed in Question 6b.
- 8) Give an explanation of the results you noted
a. when ethyl alcohol, $\text{C}_2\text{H}_5\text{OH}$, was added.
b. when the solution of aqueous ammonia, $\text{NH}_3(\text{aq})$, was added.
- 9) On the basis of your conclusion made in Question 7, predict some additional substances which in solution might have the same effect on the $\text{CrO}_4^{2-}-\text{Cr}_2\text{O}_7^{2-}$ equilibrium as those you listed in Questions 6a and 6b.



Experiment 31

Heat of Acid-Base Reactions

In Experiment 26 you studied the heat of reaction of a base, sodium hydroxide, NaOH, with hydrochloric acid, HCl. In this experiment you will compare the heat energy involved when other acids and bases react.

Are all acid-base reactions exothermic? Do some acid-base reactions release more heat energy than others, per mole of H^+ reacted? Are all solutions of acids and bases good conductors of electricity (strong electrolytes)? As you carry out this experiment you will be seeking answers to these questions.

Your teacher will assign you one or two of the twelve acid-base combinations shown below. Record your data first in your laboratory notebook and then make them available to the rest of the class, as directed by your teacher.

Combinations of Solutions to Be Tested
(Use 50 ml of each member of the pair.)

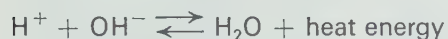
	2.0 M NaOH	2.0 M KOH	2.0 M $\text{NH}_3(\text{aq})$
2.0 M HCl	1	2	3
1.0 M H_2SO_4	4	5	6
2.0 M HNO_3	7	8	9
2.0 M CH_3COOH	10	11	12

PROCEDURE

- Use a clean Styrofoam cup as the calorimeter. Obtain 50 ml of one solution in a beaker and 50 ml of the other in the Styrofoam cup. Note the initial temperature of each solution and record it to the nearest 0.1°C . Be sure to rinse and dry your thermometer before transferring it from one solution to another.
- Quickly pour the solution from the beaker into the other solution in the Styrofoam cup. Record the highest temperature reached by the solution.
- Your teacher will demonstrate the electrical conductivity of each acid and base. Record the results in your notebook. Classify each solution as a good conductor (strong electrolyte) or as a poor conductor (weak electrolyte).

PROCESSING THE DATA

- 1) Write net ionic equations for each of the twelve reactions. Write the formulas for the strong electrolytes in ionic form and the formulas for the weak electrolytes in molecular form to indicate the reacting species present. Include ΔH for each reaction. Review the calculations you made in Experiment 26 to determine the molar heat produced by the reaction



Make a similar calculation to obtain the heat energy involved *per mole* of H^+ used in each reaction you performed in this experiment. Neglect any energy lost to the environment.

- 2) Using class data, what regularities do you observe in the ΔH values? What do all the equations have in common? Base your generalization on averages of class data.
- 3) Propose an explanation for the differences and similarities in the ΔH values based upon the energy needed to make and break chemical bonds.



Experiment 32

Acid-Base Titration

Amounts of reactants and products in a reaction are commonly investigated in two ways in the laboratory : gravimetrically (by mass), as in Experiments 6 through 9, and volumetrically (by volume and concentration), as in this experiment. **Titration** is the name given to the process used to determine the volume of a solution needed to react with a given mass, or volume, of a sample. We shall use this process to study quantitatively the reaction between an acid and a base. The hydrogen ion from the acid reacts with the hydroxide ion from the base to produce water.

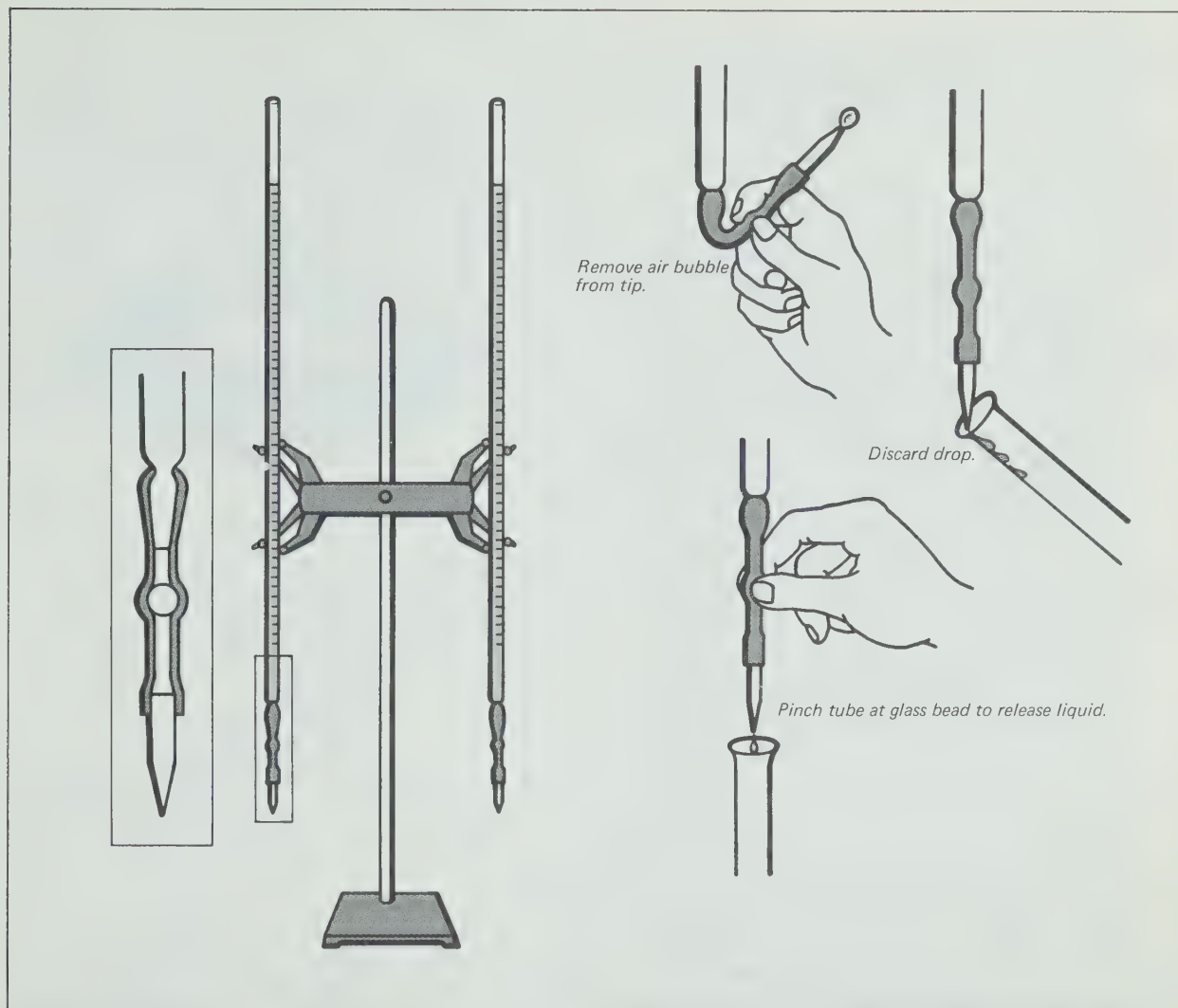
Phenolphthalein will be used as the indicator in this experiment, since its color change occurs when approximately the same number of moles of hydrogen ion and moles of hydroxide ion have been added. This point in the reaction is called the equivalence point.

PROCEDURE

Using hydrochloric acid of known concentration, you will first standardize a sodium hydroxide solution, that is, determine its concentration expressed as moles per liter. With this standard base, you will then titrate a known mass of an unknown solid acid, and then calculate the number of grams of this acid that will react with one mole of the base. After this experimental value has been determined, your teacher will tell you the formula of the acid. You will then be able to write the equation for the reaction, calculate the value for the number of grams of acid that will react with one mole of the base, and compare it with your experimental value.

Part I. Standardization of the Solution of a Base

- Obtain two burets. See the section on the care of burets on page 142. Clean the burets and rinse one with a few ml of the hydrochloric acid which you will be using in it. Rinse the other with a few ml of the sodium hydroxide solution you will be using in it. After rinsing the burets, fill the first with the acid of known concentration and the second with the base. See Figure 32-1.
- Record the volume in each buret by reading the bottom of each meniscus to the nearest 0.01 ml. Let 10 ml of hydrochloric acid flow into a clean 250 ml Erlenmeyer flask. Add 15 ml of distilled water and 3 drops of phenolphthalein.

**Figure 32-1**

Buret assembly and use.

- c. Hold the neck of the Erlenmeyer flask with one hand and manipulate the buret with the other. As you add the sodium hydroxide, gently swirl the flask so the solutions will become mixed. Continue adding sodium hydroxide until the first faint pink color develops. If the color disappears upon mixing the solution, add more sodium hydroxide, drop by drop, until a pink color persists for at least 10 seconds. Observe the volumes of acid and base used. Then

add the solution you used most of until the level drops to the 22 ml mark. Now, add the other solution until a color change occurs. Continue to add one solution drop by drop, and then the other until one drop of base gives a pink color that persists for at least 10 seconds. Take care not to go beyond the last calibration marks on the burets. Record the volume reading at the bottom of the meniscus in each buret.

- d. Refill each buret with the proper solution. Rinse the Erlenmeyer flask thoroughly. Repeat steps b and c. Do a total of three titrations.

Part II. Titration of an Unknown Acid

- e. Obtain a solid unknown acid from your teacher. Weigh the vial containing the sample to the nearest 0.01 gram. Remove a suitable amount (about one-half gram, or as directed by your teacher) of the solid acid into a clean flask as shown in Figure 32-2. Weigh the vial and contents again. Dissolve the sample in 50 ml of distilled water and add 3 drops of phenolphthalein. If all of the acid does not dissolve at this point, it will dissolve later during the titration when the acid will be converted to the more soluble sodium salt.
- f. Refill the proper buret with some of the solution of base used previously and record the initial reading. Add the base to the acid solution until a faint pink color persists at least 10 seconds. Be careful not to overrun the equivalence point. If you pass the equivalence point, add a little more of the solid acid and reweigh the vial. Be sure to include the mass of all solid acid used in each titration. Titrate to the equivalence point and record the final buret reading.
- g. Perform the titration with two more samples. Use the knowledge you gained in the first titration; that is, assuming you used 20 ml of base to titrate a certain mass of acid, and that you have almost the same mass of acid for the second trial, you can run 15 ml of base into the flask rapidly and complete the last part of the titration cautiously.

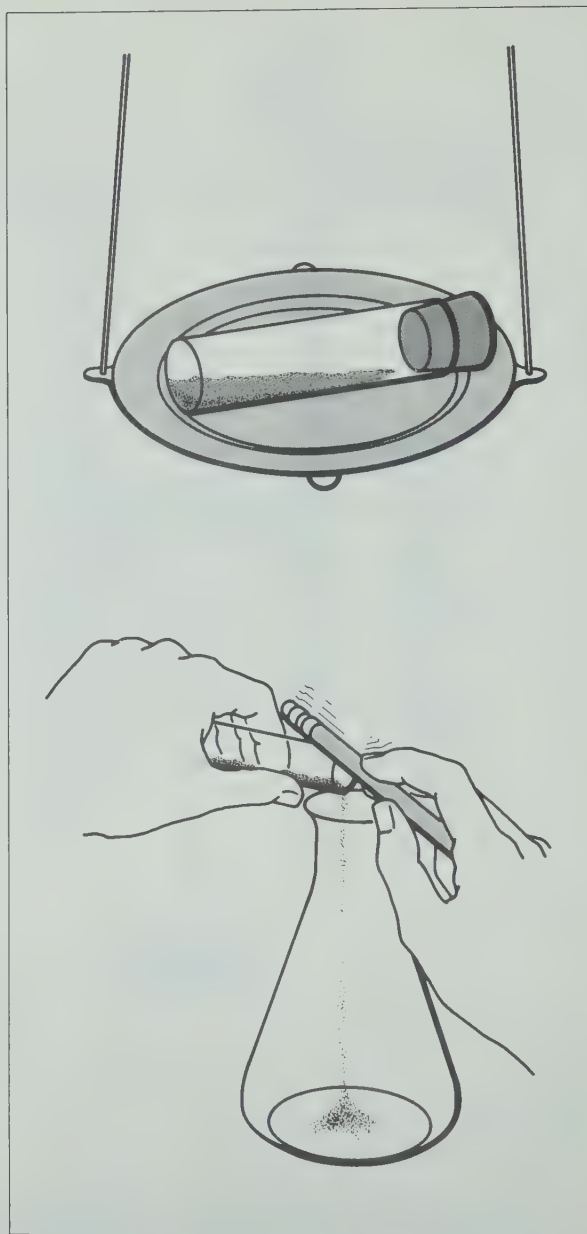


Figure 32-2

Weighing a sample and transferring it to a flask.

Part III. Optional Titration

If time permits, you may bring from home commercially available household acidic or basic substances to titrate with either your standard acid or base. Examples of items readily available are lemon juice, vinegar, household ammonia, washing powders, and Tums. Determinations which are possible are the percent acetic acid, CH_3COOH , in vinegar; the percent citric acid, $\text{C}_6\text{H}_8\text{O}_7$, in lemon juice; the percent ammonia, NH_3 , in household ammonia. Your teacher will tell you how large a sample to use and which indicator is appropriate.

PROCESSING THE DATA

- 1) From the concentration given and volume used, calculate the number of moles of hydrochloric acid involved in each titration of Part I.
- 2) Use the equation for the reaction to determine how many moles of base are used per mole of acid in Part I.
- 3) Using the relation in Question 2, determine the moles of base used in each trial.
- 4) Calculate the molarity of the base. Compute the average of the molarities found for each of the three trials.
- 5) For each trial in Part II, calculate the mass of the solid unknown acid that will react with one mole of the base. Determine the average of these values.
- 6) Using the formula of the acid given by your teacher, calculate the theoretical value for the mass of the acid that will react with one mole of the base.
- 7) Determine the percent error, using the value calculated in Question 6 as the accepted value.



Experiment 33

Determination of $[H^+]$ Using Indicators

Acid-base indicators are dyes whose colors depend upon the hydrogen ion concentration of a solution. Many dyes are suitable for this purpose, and each changes color over a particular range of hydrogen ion concentrations. You are already familiar with one dye, litmus, which is red when the $[H^+] = 10^{-6} M$ or greater and blue when the $[H^+] = 10^{-8} M$ or less.

For this experiment you will use two indicators which have color changes in acidic solutions (methyl orange and orange IV) and two indicators for basic solutions (indigo carmine and alizarin yellow R).

A series of standard acidic solutions with known concentration of $H^+(aq)$ will be made by diluting a $0.1 M$ HCl *strong* acidic solution. In a similar manner a series of basic solutions will be prepared by diluting a $0.1 M$ NaOH *strong* basic solution. These prepared solutions will serve as standards. Using appropriate indicators, you will be able to determine the $[H^+]$ of an "unknown" solution by comparing the dye color produced in the unknown solution with that formed in the standard solutions. You will also determine the hydrogen ion concentration of a solution of a *weak* acid and calculate its equilibrium constant. Finally an indicator will be used to compare the volume of $0.1 M$ NaOH required to react with equal volumes of $0.1 M$ HCl and $0.1 M$ acetic acid, CH_3COOH .

You will work in pairs during Parts I and II of this experiment. Student A will prepare the standard acidic solutions, and Student B will do the same for the basic solutions. Label the test tubes so that each of you can use the standards to determine the $[H^+]$ of an unknown solution in Part III.

PROCEDURE

Part I. The Preparation of Standard Acidic Solutions

$$[H^+] = 10^{-1} M \text{ to } 10^{-4} M$$

Student A

- Obtain about 10 ml of $0.1 M$ HCl in a clean, dry 13×100 mm test tube. Label this test tube $[H^+] = 0.1 M$. Since hydrochloric acid is a strong acid, it can be assumed to be completely ionized in this dilute solution.
- Prepare some $0.01 M$ HCl by diluting one volume of $0.1 M$ HCl with nine volumes of distilled water. Use a medicine dropper and a 10 ml graduated cylinder. An appropriate volume to use would be 1.0 ml of $0.1 M$ HCl and 9.0 ml of distilled water. Thoroughly mix this solution and label it $[H^+] = 0.01 M$ or $10^{-2} M$.
- In a similar manner prepare 10 ml of $0.001 M$ HCl by diluting some of the standard solution prepared in step b. Mix it thoroughly and label it $[H^+] = 0.001 M$ or $10^{-3} M$.

- d. Finally prepare 10 ml of 0.0001 M HCl by diluting some of the standard solution prepared in step c. Mix it thoroughly and label it $[H^+] = 0.0001 M$ or $10^{-4} M$.
- e. Pour half of each of the solutions prepared into clean test tubes so that you will have two sets of standards. Label the new tubes appropriately. To each of the four test tubes of one set add one drop of orange IV indicator solution. To each of the four test tubes of the other set add one drop of methyl orange indicator solution.
- f. Record the colors observed in each of the solutions for the various hydrogen ion concentrations. Retain these standards for Parts III and IV.

Part II. The Preparation of Standard Basic Solutions

$$[OH^-] = 10^{-1} M \text{ to } 10^{-4} M$$

Student B

Follow the directions of steps a, b, c, and d; but to prepare the basic solutions use 0.1 M NaOH instead of 0.1 M HCl. Since sodium hydroxide is a strong electrolyte, a dilute solution can be considered to be completely ionized so that the hydroxide ion concentration of 0.1 M NaOH solution is 0.1 M or $10^{-1} M$. Label the solutions thus prepared in steps b, c, and d, $[OH^-] = 10^{-2} M$, $10^{-3} M$, and $10^{-4} M$, respectively.

- g. Divide the solutions to obtain two sets of the four standards as outlined in step e. To each of the four test tubes of one set add one drop of indigo carmine solution. To each of the four test tubes of the other set add one drop of alizarin yellow R solution.
- h. Record the colors observed in each of the solutions for the various hydroxide ion concentrations. Retain these standards for use in Part III.

Part III. The Determination of the Hydrogen Ion Concentration of an Unknown Solution. Work independently.

- i. Obtain about 5 ml of an unknown solution from your teacher. Test a portion of it with litmus paper to determine the approximate hydrogen ion concentration.
- j. Place about 2 ml of the unknown solution into each of two small test tubes. If the solution is acidic, add a drop of orange IV solution to one test tube and a drop of methyl orange to the other test tube. If the solution is basic, use indigo carmine and alizarin yellow R.
- k. Compare the colors with those of the standards prepared by either you or your partner. Record the **hydrogen ion** concentration of the unknown solution.

Part IV. The Determination of the Hydrogen Ion Concentration of a Solution of a Weak Acid

- l. Obtain about 5 ml of a solution of acetic acid in a small, clean, dry test tube. Note and record the concentration. Those students who prepared acid standards will use a 0.1 M solution, and others will use a 1.0 M solution.
- m. Place about 2 ml of the acetic acid solution into each of two small, clean, test tubes. Add a drop of orange IV solution to one test tube, and a drop of methyl orange solution to the other.
- n. Compare the colors with those of the standards containing the same indicators and estimate the $[H^+]$ of the acid solution as accurately as you can.

Part V. The Determination of the Volume of 0.1 M NaOH Required to React with Equal Volumes of 0.1 M HCl and 0.1 M CH_3COOH

- o. Use a clean medicine dropper to measure out 20 drops of 0.1 M HCl solution into a clean, small test tube.

- p. Rinse the medicine dropper thoroughly and measure out 20 drops of 0.1 *M* CH₃COOH solution into another test tube.
- q. Add a drop of phenolphthalein indicator to each test tube. Phenolphthalein is a dye which is colorless in the range where the $[H^+] = 10^{-1} M$ to $10^{-9} M$, but turns pink when the $[H^+]$ is less than $10^{-9} M$ ($[OH^-]$ is greater than $10^{-5} M$).
- r. Rinse the medicine dropper thoroughly and add 0.1 *M* NaOH solution a drop at a time to each acidic solution until the indicator turns to a pink color which does not disappear on mixing the solution in the test tube. Record the number of drops of 0.1 *M* NaOH required for each solution.
- 2) Predict qualitatively the effect of each of the following experiments on the acetic acid equilibrium reaction:
- Some of the salt sodium acetate, which produces the ions Na⁺(aq) and CH₃COO⁻(aq), is dissolved in 0.1 *M* acetic acid solution. Would the $[H^+]$ increase or decrease?
 - Some sodium hydroxide solution is added, drop by drop, to 0.1 *M* CH₃COOH solution.
- 3) Explain the results obtained in Part V, where you compared the number of drops of 0.1 *M* NaOH solution required to reach the phenolphthalein endpoint, using equal volumes of 0.1 *M* HCl and 0.1 *M* CH₃COOH solutions.

PROCESSING THE DATA

- 1) Calculate the equilibrium constant for the equilibrium reaction involving an aqueous solution of the weak acid, acetic acid.



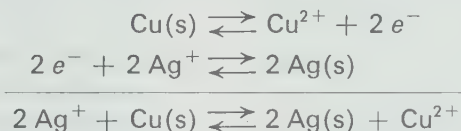
Use the value for the hydrogen ion concentration determined in step I. You may assume that the concentration of the acetate ion, CH₃COO⁻, is equal to the $[H^+]$ and that the concentration of acetic acid is essentially the concentration of the solution you used, either 0.1 *M* or 1.0 *M*.

- 4) Calculate the $[H^+]$ of each of the solutions prepared in Part II. Use the relation $[H^+][OH^-] = 10^{-14}$.
- 5) Calculate the pH of each of the solutions prepared in Parts I and II.
- 6) Determine the $[H^+]$ of your unknown solution.
- 7) The H⁺(aq) concentration of a 1 *M* solution of benzoic acid is $8 \times 10^{-3} M$.
- What percent of the benzoic acid, C₆H₅COOH, is ionized in this aqueous solution?
 - Predict what volume of 1 *M* NaOH would be required to neutralize 10 ml of 1 *M* benzoic acid.

Experiment 34

An Introduction to Oxidation-Reduction

In Experiment 7 you observed the reaction in which copper metal was oxidized to cupric ions by silver ions. In the same reaction, the silver ions were reduced to silver metal. In that example there was a transfer of electrons from Cu(s) to $\text{Ag}^+(\text{aq})$. The Cu(s) acted as the reducing agent. It reduced the $\text{Ag}^+(\text{aq})$ ion. The $\text{Ag}^+(\text{aq})$ ion acted as the oxidizing agent. It oxidized the Cu(s) .



In Part I of this experiment you will observe some possible oxidation-reduction reactions involving several metals and metallic ions. By analyzing your results you can determine the relative strengths of the metals as reducing agents (tendency to lose electrons) and of the metallic ions as oxidizing agents (tendency to gain electrons).

In Part II you will make a similar comparison of the relative oxidizing strengths of three members of the halogen family: chlorine, bromine, and iodine. You will determine which halogen, Cl_2 , Br_2 , or I_2 , is capable of removing electrons from the halide ions, Cl^- , Br^- , and I^- . This information will help you arrange the halide ion-halogen element half-reactions, $2\text{X}^- \longrightarrow \text{X}_2 + 2e^-$, in order of decreasing ease of oxidation.

PROCEDURE

Part I

- Obtain small, clean strips of the metals zinc, copper, and lead. Also have available the following 0.1 *M* solutions: $\text{Zn}(\text{NO}_3)_2$, $\text{Cu}(\text{NO}_3)_2$ and $\text{Pb}(\text{NO}_3)_2$.
- Allow each of the metals to come into contact with each of the solutions. For every combination use 3 ml of the solution in a 13×100 mm test tube and a freshly cleaned strip of metal.

- Record the combinations which produced a reaction. Briefly describe the evidence for the reaction. Record whether a large, medium, or small amount of new substance was formed.

Part II. Preliminary Observations

- Obtain a small (pin-head size) crystal of $\text{I}_2(\text{s})$ in each of two small test tubes. Add 2 ml of H_2O to one and 2 ml of trichlorotrifluoroethane (TTE) to the other. Cover with a plastic square, and shake each tube.

Record observations. In which solvent is $I_2(s)$ more soluble?

- e. To the tube containing $I_2(s)$ and water, add 1 ml of TTE. Observe whether the TTE sinks or floats on the water. Cover and shake the tube. Record observations.
- f. To a tube containing 3 ml of 0.1 *M* KI solution, add 1 ml of TTE. Cover and shake the tube. Record observations. Do both I^- and I_2 react the same in TTE?
- g. Obtain 3 ml of an aqueous solution of one of the three halogens in each of 3 separate test tubes. The iodine solution contains a little ethanol. This increases the solubility of iodine. To each, add 1 ml of TTE, cover, and shake the tube. Record observations.

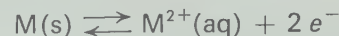
Test for Spontaneous Oxidation-Reduction Reactions

- h. Put 3 ml of 0.1 *M* NaBr in one test tube and 3 ml of 0.1 *M* NaI in another. To each tube add 1 ml of TTE and 1 ml of chlorine water. Cover and shake each tube for 15 seconds. Note the color of the TTE phase and compare with the preliminary tests in step g.
- i. Repeat the test outlined in step h, except use 0.1 *M* NaCl and 0.1 *M* NaI. Add 1 ml TTE and five drops of bromine water to each. Shake the tubes as directed above. Record your results.
- j. Repeat the test outlined in step h, except use 0.1 *M* NaCl and 0.1 *M* NaBr. Add 1 ml TTE and five drops of the iodine solution to each. Shake the tubes as directed before. Record your results.

PROCESSING THE DATA

- 1) Which of the metals tested was oxidized by both of the solutions of the other metallic ions? Which one was oxidized by only one of the metallic ions? Which one was oxidized by neither of the other metallic ions?

- 2) Compare the metal-metal ion pairs by arranging their half-reactions



in a column in order of decreasing ease of oxidation. Since you know from Experiment 7 that $Cu(s)$ was oxidized by $Ag^+(aq)$, add the $Ag(s) \rightleftharpoons Ag^+(aq) + e^-$ half-reaction to your list in the appropriate place.

- 3) Write balanced equations for the cases where oxidation-reduction reactions between metals and metallic ions were observed [see the example for $Cu(s)$ and $Ag^+(aq)$ in the introductory section of this experiment].
- 4) Which of the halide ions tested was oxidized by both of the other halogen elements? Which halide ion was oxidized by only one halogen element? Which halide ion was not oxidized by any of the halogen elements used?
- 5) Compare the halide ion-halogen element pairs by arranging their half-reactions in a column in order of decreasing ease of oxidation.
- 6) Write balanced equations for the cases where oxidation-reduction reactions occurred between halide ions and elementary halogens.
- 7) Use the additional information given below to construct a series of all seven half-reactions discussed in this experiment in order of decreasing ease of oxidation.
 - a. $Ag^+(aq)$ is a stronger oxidizing agent than $I_2(s)$ but weaker than $Br_2(l)$.
 - b. $I^-(aq)$ is a weaker reducing agent than is $Cu(s)$ but is a stronger one than $Ag(s)$.
- 8) Would it be feasible to store a solution of copper sulfate in a container made of metallic zinc? Of metallic silver? Give reasons for your answers.

Experiment 35

Corrosion of Iron

Each year industry in the United States alone loses billions of dollars as a result of the corrosion of iron. What are some of the factors which are responsible for this loss? What can be done to reduce it?

Corrosion is a general term applied to the process in which metals are converted to oxides or other compounds. This causes the gradual deterioration of metals. Although the detailed chemistry of the corrosion of iron is not completely understood, it clearly involves oxidation. In this experiment we shall investigate some of the factors involved in corrosion and try to relate them by some generalizations.

PROCEDURE

Part I. Reaction of Iron with Various Aqueous Reagents

- Place a clean, bright nail in each of five test tubes. Slide each nail carefully down the side to avoid breaking the bottom of the tube.
- Partially fill each of the test tubes with one of the following reagents so that the nail is just covered. Your teacher will tell you which one of the groups to use. All solutions are 0.1 *M*.

Group A	Group B	Group C
NaOH	KOH	Na_3PO_4
$\text{Na}_2\text{Cr}_2\text{O}_7$	Na_2CO_3	$\text{Na}_2\text{C}_2\text{O}_4$
NaCl	KNO_3	NaSCN
HCl	HNO_3	H_2SO_4
H_2O	H_2O	H_2O

- Determine whether the solution is acidic, basic, or neutral, using litmus or Hydrion paper. Allow the nails to stand overnight in the solutions. Go on to Part II. After the solutions have been standing overnight, observe and record any changes that have taken place. To each solution add one or

two drops of 0.1 *M* potassium ferricyanide solution, $\text{K}_3\text{Fe}(\text{CN})_6$, which contains the ions $\text{K}^+(\text{aq})$ and $\text{Fe}(\text{CN})_6^{3-}(\text{aq})$. Observe any change. Compare your results with those of students using the other sets of reagents. Record their observations with yours in tabular form in your notebook.

- Add one drop of 0.1 *M* potassium ferricyanide solution to about 1 ml of ferrous sulfate solution. Compare this result to that obtained when the potassium ferricyanide was added to the various solutions containing nails. What conclusions can be made from the results in step c?

Part II. Reactions Involving Two Metals

- Prepare about 200 ml of agar mixture as follows: heat about 200 ml of distilled water to a gentle boil. Remove the burner and stir in 2 grams of powdered agar. Resume heating and stir until the agar is dispersed.
- Add about 10 drops of 0.1 *M* potassium ferricyanide and 5 drops of 0.1% phenolphthalein solution to the agar mixture. Stir thoroughly.

- g. While the agar mixture is cooling, prepare four clean, bright nails as shown in Figure 35-1. Place one nail on one side of a petri dish or small beaker. Bend another nail sharply with a pair of pliers and place it on the other side of the dish. Twist a clean piece of bare copper wire around a third nail. Then remove the nail and tighten the wire coil so that when the nail is forced back through it makes tight contact with the wire. Place this in a second dish. Repeat the above procedure, using a zinc strip on a fourth nail. If a strip of zinc may be attached by forcing the nail through the zinc piece in at least two places. Place this nail in the second petri dish as shown in Figure 35-1. Be sure the nails do not touch one another.

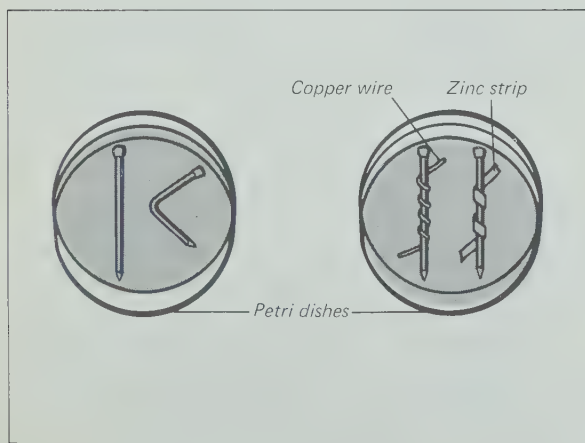


Figure 35-1

Fill dishes half full of agar (transparent gelatin) covering nails.

- h. When the agar mixture has cooled to lukewarm and is still fluid, pour it carefully into the petri dishes until the nails and attached pieces of metal are covered to a depth of about 0.5 centimeter.

- i. Make observations during the time remaining in the class period. Place the dishes in your locker and observe them again after they have stood overnight.

PROCESSING THE DATA

- 1) List the reagents in Part I in which no indication of corrosion was observed.
- 2) List the reagents in Part I in which there was an indication of corrosion.
- 3) Are any regularities in evidence? What is the evidence? How do you account for the regularity?
- 4) What did you observe regarding the reactions at the head, the pointed end, or the sharp bend of the nail which were different from the rest of the nail? Account for this in terms of the mechanical treatment of the nail during its manufacture.
- 5) Ferrous ions react with potassium ferricyanide to form a colored precipitate. Write an equation for the reaction.
- 6) Which color in Part II indicates the site of oxidation? Which, the site of reduction? Account for the formation of each color.
- 7) Write the oxidation and reduction reactions for each experiment in which you observed a reaction in Part II.
- 8) Consult the table in Appendix 4 and predict another metal that is more readily oxidized than iron and will protect it from corrosion. You may want to test your prediction by an experiment.
- 9) How does a coating of zinc on iron (galvanized iron) protect iron from corrosion?
- 10) Magnesium metal rods are sometimes placed in hot water heaters. Why?



Experiment 36

Moles of Copper, Moles of Silver, and Moles of Electrons Involved During Electrolysis

Michael Faraday was the first person to relate amount of electricity to amount of matter formed at or removed from electrodes during electrolysis. His work was done about 1830. In this experiment, we will interpret "amount of electricity" in terms of number of electrons. Faraday could not do this because the electron was not discovered until 1897.

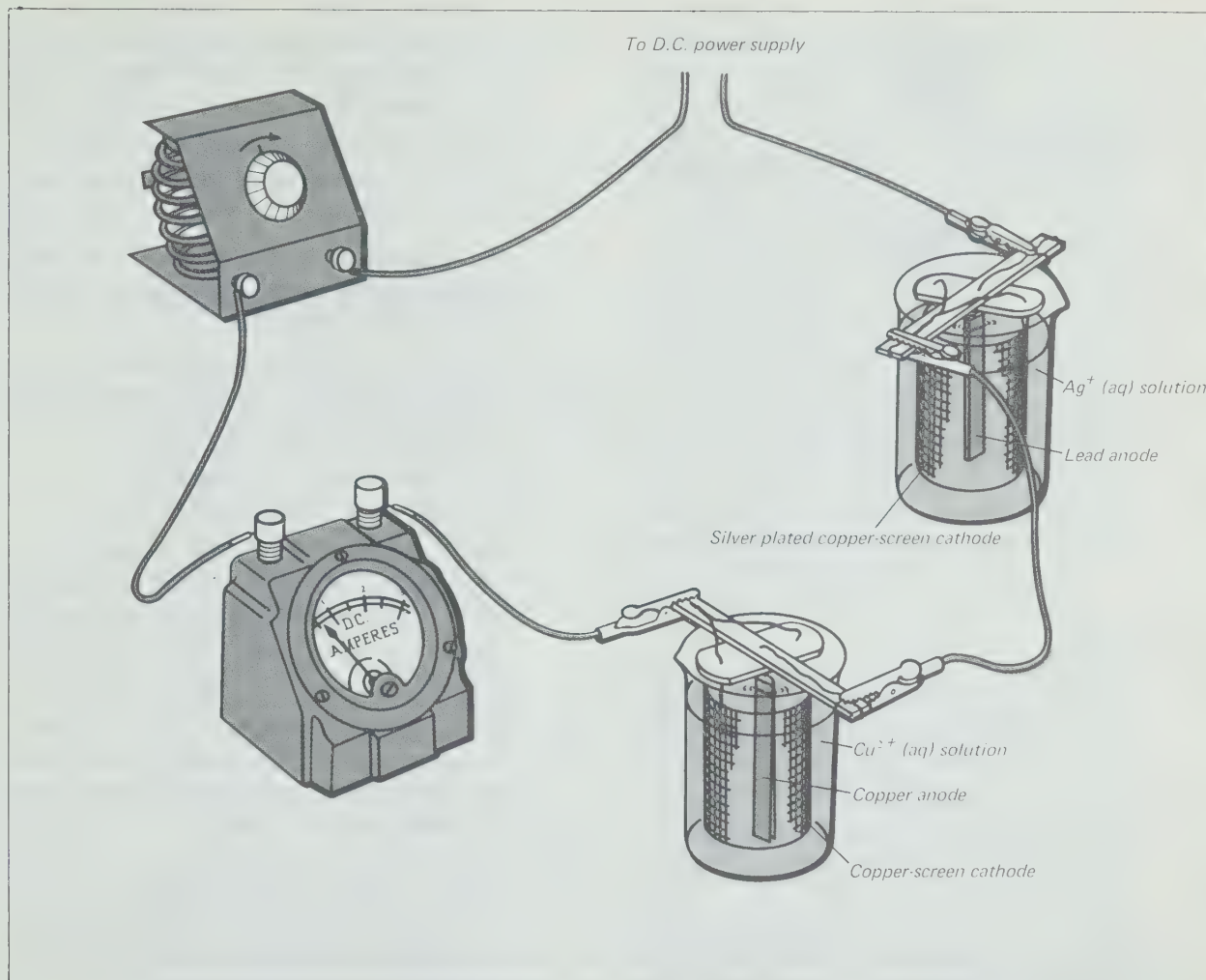
Figure 36-1 shows the details of the electric circuit and the cells we shall use. The source of electrons is either a battery or another suitable source of direct current which is constructed such that the electrons are made to flow in one direction. The ammeter measures the rate of flow of electrons (the current), passing through the circuit in a given unit of time. Since electric charge does not accumulate in any one part of the circuit, the rate of flow of electrons through all parts must be the same. One ampere is the current flow when 1.04×10^{-5} mole of electrons passes through the conductor during one second.

The source of direct current causes the electrons to enter one terminal or electrode and to leave the other. The circuit is complete when at one electrode in each solution a molecule accepts electrons and is reduced, while at the other electrode a molecule loses electrons and is oxidized. The electrode at which reduction occurs is the cathode and that at which oxidation occurs is the anode.

The materials and circuit shown in Figure 36-1 will be used to determine the relation between the number of moles of electrons flowing during the experiment, the number of moles of copper ions reduced, and the number of moles of silver ions reduced.

PROCEDURE

- a. Obtain a cylinder of copper screen to be used as the cathode and a sheet of copper to serve as the anode. Clean the electrodes as directed by your teacher. Handle the electrodes by the connecting wires only. Otherwise, your fingers may leave grease marks upon which the copper deposit will not adhere well.
- b. Obtain another cylinder of copper screen. This one should have a coating of silver on it and is to be used as a cathode. A strip of lead will serve as the anode in the silver plating cell. Do not touch the clean electrodes with your fingers.
- c. Weigh each of the clean, dry cathodes (screen cylinders) to the nearest 0.01 gram.

**Figure 36-1**

Two electrolytic cells in a series circuit.

- d. Suspend the electrodes in separate 400 ml beakers as directed by your teacher. Be sure that the anodes are centered within the cylindrical cathodes and that the electrodes do not touch each other.
- e. Make all of the connections in the circuit as shown in Figure 36-1, EXCEPT leave one of the wires to the variable resistor disconnected. Adjust the variable resistor so that its full resistance will be used. Ask your teacher to check your setup.
- f. Add the solution containing the copper ions to the beaker with the copper anode. Use enough solution to reach nearly to the top of the copper-screen cathode.
- g. In a similar manner add the silver plating solution to the other beaker.
- h. Make the last connection and *quickly* adjust the current to 1.0 ampere by decreasing the resistance being used in the variable resistor. Record the starting time to the nearest second.

- i. Let the current flow for 30.0 minutes. Watch the ammeter and keep the current as close to 1.0 ampere as possible by adjusting the variable resistor.
 - j. Record the time to the nearest second as you disconnect one of the wires to break the circuit. Rinse the two cathodes by gently dipping each into a beaker containing some cold water. Do not agitate the electrodes so vigorously that the metal deposits are dislodged.
 - k. Rinse the cathodes with acetone to remove the water droplets. Allow them to dry by evaporation for about 2 or 3 minutes.
 - l. When thoroughly dry, each of the cathodes should be weighed to the nearest 0.01 gram on the same balance you used before.
- 4) Calculate the ratio between moles of electrons and moles of copper formed. Use your answers to Questions 1 and 2.
 - 5) Calculate the ratio between moles of electrons and moles of silver formed. Use your answers to Questions 1 and 3.
 - 6) Write equations for the cathode reactions. Assume that the reacting species are simple copper ions or silver ions.
 - 7) Calculate the ratio between moles of silver formed and moles of copper formed. Use your answers to Questions 2 and 3.
 - 8) How does this ratio compare to the ratio you obtained in Experiment 7?
 - 9) Write the balanced equation for the oxidation reaction which occurs at the copper anode. How would you expect the loss of mass at the copper anode to compare with the gain of mass at the copper cathode?

PROCESSING THE DATA

- 1) Calculate the number of moles of electrons which were used. Recall that 1.04×10^{-5} mole of electrons is involved when a current of one ampere flows for one second.
 - 2) Calculate the number of moles of copper deposited on the cathode from the solution containing copper ions.
 - 3) Calculate the number of moles of silver deposited on the cathode from the solution containing silver ions.
- 10) What are some sources of error in your measurements and procedures? How many significant figures in the mole relations can be justified by your data?
 - 11) How many moles of electrons would be required to plate 52.0 g of chromium on cathode from a suitable cell containing an electrolyte solution in which the chromium has an oxidation number of +6?



Experiment 37

Reactions Between Ions in Solution

The solubility table in Appendix 5 permits you to predict which ions in aqueous solutions combine to produce a precipitate of a slightly soluble compound. The oxidation-reduction table in Appendix 4 compares the relative strengths of oxidizing and reducing agents and permits you to predict when certain ionic species in solution will undergo an oxidation-reduction reaction. Remember that predictions based on E° values show only which is favored, the reactants or the products, after equilibrium is reached. They do not predict the rate of the reaction. In cases where the reaction you predicted does not occur immediately, make observations again later in the period or the next day before you evaluate your predictions.

In Part I of this experiment you are to predict, before coming to the laboratory, whether an appreciable reaction will take place when each of the ten combinations of solutions of ions are mixed. If you predict that a reaction will take place, write a balanced equation for it in your laboratory notebook. Use ions where appropriate, and label precipitates with (s) and gases with (g).

Check your predictions by performing the experiments in the laboratory. Include those for which you predicted no reaction. Do not be biased by your predictions; record what you actually observe. For those observations not in agreement with prediction, write equations representing your observations.

In Part II you need not make preliminary predictions. These reactions are somewhat more complex than the ones in Part I. Record your observations as you perform the experiments, and later attempt to explain the results. Write balanced ionic equations for the reactions you can interpret.

PROCEDURE

Part I. Predict the Results

For each trial use about 3 ml portions of each solution and mix them thoroughly in a small test tube. All solutions are 0.1 *M* concentration unless otherwise specified.

- magnesium nitrate and sodium hydroxide
- magnesium nitrate and sodium sulfate
- barium hydroxide (saturated) and sulfuric acid
- potassium dichromate and sodium sulfite (acidified with 1 drop of 6 *M* sulfuric acid) First add the acid to the sodium sulfite solution. Note any odor. Then add the potassium dichromate solution.
- 0.05 *M* potassium permanganate and 1.0 *M* hydrochloric acid Observe again after 10 minutes.
- potassium iodide and ferric chloride Add about 1 ml of TTE (trichlorotrifluoroethane) to the mixture and shake the test

tube. This makes it easier to detect the presence of any halogen, owing to its greater solubility in the TTE phase. Review the solubility of iodine in TTE as used in Part II of Experiment 34.

- g. potassium bromide and ferric chloride
Add 1 ml of TTE and shake the test tube.
- h. ferrous sulfate (acidified with 3 drops of 6 *M* sulfuric acid) and 0.01 *M* potassium permanganate
- i. zinc sulfate and ammonium carbonate
- j. barium hydroxide (saturated) and zinc sulfate

Part II. Other Reactions

The following reactions are more complex than those in Part I. You are not expected to make predictions. Do the experiments, make careful observations, and later attempt to interpret the results. Write ionic equations for the reactions you can interpret.

Experiments k through n are a series involving reactions of hydrogen peroxide and some ions of chromium in both acidic and basic solutions.

Use 3 ml of each solution and mix thoroughly.

- k. Acidify 0.1 *M* chromium sulfate with 3 drops of 6 *M* sulfuric acid. Add 3% hydrogen peroxide.
 - l. Add 6 *M* sodium hydroxide drop by drop to 0.1 *M* chromium sulfate until the precipitate, $\text{Cr}(\text{OH})_3$, first formed dissolves producing $\text{Cr}(\text{OH})_4^-$ ions. Add 3% hydrogen peroxide.
 - m. Acidify 0.1 *M* potassium dichromate with 3 drops of 6 *M* sulfuric acid. Add 3% hydrogen peroxide.
 - n. Add 6 *M* sodium hydroxide drop by drop to 0.1 *M* potassium dichromate until the solution turns yellow. Add 3% hydrogen peroxide.
- Experiments o through q are a series involving reactions of lead ion with various anions.
- o. Mix 3 ml of 0.1 *M* lead nitrate with 3 ml of 0.1 *M* sodium chloride.
Allow the precipitate to settle a few minutes and decant about 3 ml of the liquid from the residue for use in step p. Some of the precipitate can be present in the decanted liquid; the liquid does not have to be clear.
 - p. Add 3 ml of 0.1 *M* potassium iodide to 3 ml of the decanted liquid from step o.
Allow the precipitate to settle and decant the liquid from the residue for use in experiment q. Some of the precipitate can be present in the decanted liquid; the liquid does not have to be clear.
 - q. Add 3 ml of 0.1 *M* sodium sulfide to 3 ml of the decanted liquid from step p.

Experiment 38

The Packing of Atoms in Crystals

The crystalline state of matter consists of a regular arrangement of atoms, molecules, or ions. If we represent each building block as a point, then the crystal structure can be represented by a regularly repeating pattern called the space lattice. In this experiment we shall use Styrofoam spheres as the building blocks, and we shall study some of the ways they can be packed to form some typical crystals. Three types of packing will be investigated: hexagonal closest packing, face-centered cubic packing (also called cubic closest packing), and body-centered cubic packing. We shall observe the number of nearest neighbors (the **coordination number**) of the particles in each of these structures.

In addition, we shall investigate one method of packing spheres of different radii in order to represent an ionic crystal.

PROCEDURE

Secure a supply of Styrofoam spheres of the following diameters: 36 2-inch and 13 1-inch. Obtain 38 pieces of chenille stems each 1 inch long for connecting the spheres.

Part I. Some General Considerations on Packing of Spheres

- Determine how many 2-inch spheres you can pack around a marked 2-inch sphere in the same plane. If all spheres are the same size, does the number of spheres contacting a central sphere depend on the size of the spheres? Check your prediction.
- Place spheres above the marked sphere so that they all touch it. Could the same situation prevail under the marked sphere? What is the maximum number of spheres of one size that can contact a central sphere? What is the coordination number for this type of closest packing?

Part II. Model A — Hexagonal Closest Packing

- Connect the groups of spheres you used in Part I with short lengths of chenille stems to obtain the layers shown in Figure 38-1.

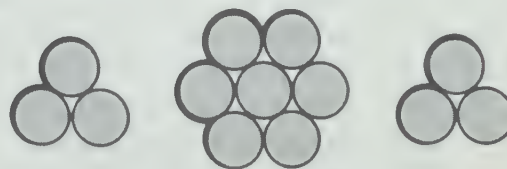


Figure 38-1

Layers for hexagonal closest packing, model A.

- Place a 3-sphere layer on the desk with the apex of the triangle facing you.
- Now place the 7-sphere layer over the 3 spheres such that the center sphere fits closely into the depression of the bottom layer.
- Center the last 3-sphere layer so it is over the central sphere of the large layer and

also directly above the spheres in the bottom layer.

If a pattern such as this were expanded into space until billions of atoms were involved, one would have a model of a very small crystal of some metal. Magnesium and zinc are examples. Note the coordination number. Retain this model for use in Part IV.

Part III. Model B — Cubic Closest Packing or Face-centered Cubic Packing

- g. Construct the layers illustrated in Figure 38-2 using 2-inch spheres and chenille stems as before.



Figure 38-2

Layers for cubic closest packing, model B.

- h. Place the first layer flat on the desk. Place the second layer on it such that the spheres rest in the spaces between the corner spheres of the first layer. Now add the third layer such that its spheres are directly over those in the first layer. Study this model carefully. Why is it called face-centered cubic? This is the packing which is found in copper, silver, aluminum, and many other metals.

Part IV. Comparison of Hexagonal Closest Packing with Cubic Closest Packing

- i. Return to the hexagonal closest packing Model A which you made in Part II. Rearrange it such that the top layer is not directly over the first layer but is rotated at 60° with respect to it.
- j. Rotate this model slightly and look for 4 spheres forming a square facing you. Now take the top layer off of Model B from Part III and place it on the 4 spheres you located on Model A. Note that this new model contains a face-centered cube, just like Model B.

- k. In the light of the above comparison, is there a difference in coordination number in the two types of closest packing? Is there a difference in density when spheres of comparable mass and size are involved in each of these types of packing? Most metals crystallize in only one, not both, of these forms. What does this indicate about the directional nature of bonds between atoms of these metals?

Part V. Model C — Body-centered Cubic Packing

- l. Construct the layers shown in Figure 38-3, using 2-inch spheres from Model A. Be sure to leave space of about $\frac{1}{4}$ inch between the spheres as indicated.



Figure 38-3

Layers for body-centered cubic packing, model C.

- m. Place the single sphere in the center of the first layer and then place the third layer such that its spheres are directly over the first layer. Study the symmetry of this model and justify its name. This type of packing is typical of the alkali metals, for example, sodium and potassium. Can you suggest any reason why Na and K crystallize in this form, while most of the other metals crystallize in a closest-packed form? (Consider the number of valence electrons.)
- n. Metallic iron crystallizes in the body-centered cubic form called α -ferrite below 906°C . Above this temperature the stable form is γ -ferrite, which is face-centered cubic. At 1401°C the crystal form changes back to a body-centered cubic solid called δ -ferrite. What is the coordination number of iron in each of these forms? Try to suggest a reason for these transitions in terms of numbers of available bonding electrons.

Part VI. The Sodium Chloride Lattice

- o. Ionic crystals are formed by packing positive and negative ions into a lattice. Sodium ions have a diameter of $1.90 \text{ \AA}$ while that of the chloride ions is $3.62 \text{ \AA}$. We shall use 1-inch spheres for Na^+ and 2-inch spheres for Cl^- in our model, and thus approximate their relative sizes.
- p. Use Model B from Part III for the face-centered cubic arrangement of the chloride ions. Now insert 13 1-inch spheres, representing sodium ions, into the holes between the chloride ions in each layer. Note that the Na^+Cl^- lattice is an interpenetrating set of face-centered cubes — one involving Na^+ and one Cl^- .
- q. What type of ion surrounds each Na^+ ? Each Cl^- ? What is the coordination number of the spheres representing Na^+ , and of those representing Cl^- ?
- r. Note that in order to achieve this type of lattice there must be a favorable relation between the relative radii of the two spheres which will permit a given sphere to fit into a given hole in the lattice. What is the radius ratio for Na^+/Cl^- ions? Can you account for the stability of this type of packing in terms of interionic forces?

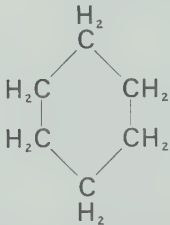
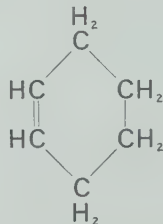
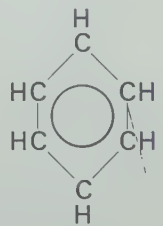
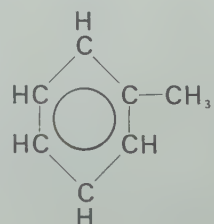
PROCESSING THE DATA

- 1) Write a brief description of each type of packing of metallic crystals that you studied.
- 2) Answer all questions raised in the Procedure Sections. Label them by Parts and Sections: for example, P I-a.
- 3) In one of the types of cubic packing, the spheres occupy about two-thirds of the space and in the other they fill about three-fourths of the space available. Identify which type is which. Which is more dense? In which does each atom form the larger number of bonds?
- 4) Suppose you have a crystal XY with the sodium chloride packing in which each of the ions is the same size as the Na^+ and Cl^- respectively but each is doubly charged X^{2+} and Y^{2-} . Would XY have a higher or lower melting temperature than NaCl ? Suggest a real pair of crystals which meets the above criteria and look up their melting temperatures to check your prediction.
- 5) Suppose you have a crystal AB with the sodium chloride packing in which each of the ions has the same charge A^+ and B^- as Na^+ and Cl^- , but the radii of A and B are proportionately larger. Would AB have a higher or lower melting temperature than NaCl ? Suggest a real pair of crystals that meets the above criteria and look up their melting temperatures to check your prediction.

Experiment 39

Some Reactions of Hydrocarbons and of Alcohols

In the first part of this experiment, you will investigate the reactivity of different kinds of hydrocarbons.

KIND OF HYDROCARBON	EXAMPLE	STRUCTURAL FORMULA
Saturated and cyclic	cyclohexane, C_6H_{12}	
Unsaturated and cyclic	cyclohexene, C_6H_{10}	
Aromatic	benzene, C_6H_6	
Aromatic and substituted	toluene, $C_6H_5CH_3$	

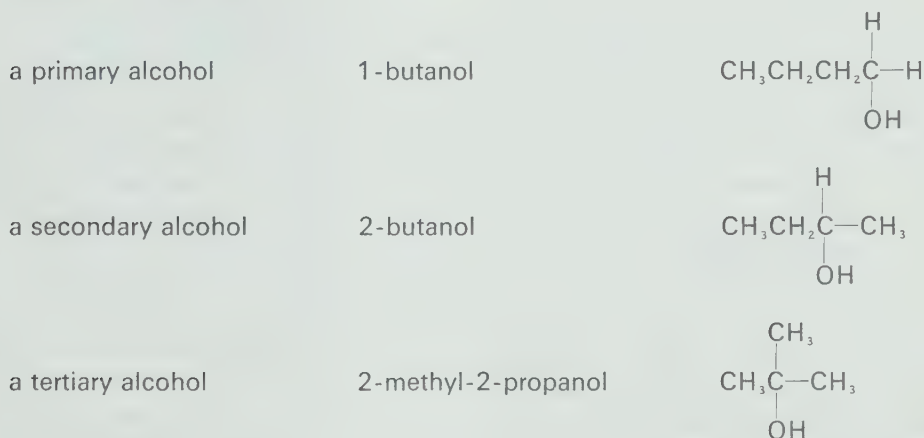
You will investigate the relative ease of oxidation of these compounds by a strong oxidizing agent, an alkaline solution of potassium permanganate. You will also compare their ability to add or substitute bromine when treated with a solution of bromine, Br_2 , in trichlorotrifluoroethane, TTE.

In the second part of this experiment, you will investigate the reactivity of some alcohols. Three of them are isomeric, each having the formula $\text{C}_4\text{H}_9\text{OH}$. It is convenient to classify alcohols by the difference in the bonding around the carbon to which the functional OH group is attached.

A **primary** alcohol has the OH group attached to a carbon which is attached to **one** carbon atom.

A **secondary** alcohol has the OH group attached to a carbon which is attached to **two** carbon atoms.

A **tertiary** alcohol has the OH group attached to a carbon which is attached to **three** carbon atoms.



PROCEDURE

Part I. Reactions of Hydrocarbons

- Label a clean, dry 13×100 mm test tube for each of the hydrocarbons to be tested: cyclohexane, cyclohexene, benzene, and toluene. Add about 10 drops of the appropriate hydrocarbon to each tube. Prepare 4 ml of a 0.005 M alkaline potassium permanganate solution by adding 2 ml of 0.01 M KMnO_4 to 2 ml of 6 M NaOH. Add 20 drops (1 ml) of this solution to each of the tubes containing the different hydrocarbons. Cover each tube with a clean plastic square and shake the contents gently to obtain more intimate contact between the two phases. Note any changes in the color of the aqueous layer after about one minute. Shake the contents occasionally and observe the tubes after five minutes.
- Place 10 drops of each hydrocarbon into four properly labeled, small test tubes. Add drops of 0.1 M Br_2 in TTE to each of the tubes. Cover each tube with a clean plastic square and shake the contents occasionally as you add the bromine solution and note any changes in color. Continue the addition of the bromine to those hydrocarbons where a change is noted until the bromine color persists, or until 20 drops have been added.

Part II. Some Reactions of Alcohols

- c. Test the reaction of ethanol, $\text{C}_2\text{H}_5\text{OH}$, with neutral, acidic, and basic solutions of potassium permanganate.

Place 2 ml of 0.01 M KMnO_4 in each of three small test tubes. Add 2 ml of distilled water to one, 2 ml of 6 M H_2SO_4 to the second, and 2 ml of 6 M NaOH to the third. Label them neutral, acidic, and basic KMnO_4 . Now add 2 drops of $\text{C}_2\text{H}_5\text{OH}$ to each. Cover with clean plastic squares and shake the contents. Note any changes in the color of the permanganate solutions. Add another drop or two of ethanol to each tube and observe any further changes which may take place after five minutes. Note any differences in the rate of oxidation as well as in the reaction products. (*Note:* The color of a solution containing the manganate ion, MnO_4^{2-} , is green; manganese dioxide, MnO_2 , is a brown precipitate; and a solution containing the manganous ion, Mn^{2+} , is very light pink, almost colorless.)

- d. The reaction of methanol, CH_3OH , with hot copper oxide is next investigated. Wrap a penny with a few turns of heavy copper wire so that it can be suspended above 10 ml of methanol in a small beaker. Place a glass stirring rod across the beaker and hook the wire over it such that the penny is suspended 1 cm above the surface of the methanol. Remove the penny and wire to a flame well away from the beaker and heat to a dull red. Quickly suspend the hot penny above the methanol in the beaker and note the interesting cyclic reaction that occurs. Should the vapor ignite, smother the flame with an asbestos square. Cautiously smell the vapors and compare them with those of methanol. The new substance formed is

formaldehyde, HCHO , which you may recognize as the liquid used to preserve specimens in the biology laboratory.

- e. Compare the following reactions of three isomeric alcohols with the formula $\text{C}_4\text{H}_9\text{OH}$.

- i) Reaction with metallic sodium.

Place 1 ml of 1-butanol in a small **DRY** test tube. Add a very small piece of freshly cut metallic sodium. Note any reaction that occurs.

Repeat this test with the other two isomeric alcohols.

- ii) Reaction with concentrated HCl .

Note: This reagent is used to compare the ease with which the $-\text{OH}$ group of the alcohol $\text{R}-\text{OH}$ reacts with 12 M HCl to form H_2O and the alkyl chloride, $\text{R}-\text{Cl}$. The alkyl halide is only slightly soluble in the aqueous phase and its presence is shown by a cloudiness due to the suspension of $\text{R}-\text{Cl}$ droplets in the water.

Place 1 ml of 1-butanol in a small test tube. Add 5 ml of 12 M HCl . Cover the tube with a plastic square. **VERY CAREFULLY** shake the mixture, and after a minute look for the presence of the slightly soluble alkyl chloride.

Repeat this test with the other two isomeric alcohols.

- iii) Reaction with a neutral solution of 0.01 M KMnO_4 .

Place 2 ml of 0.01 M KMnO_4 solution in a small test tube. Add an equal volume of 1-butanol, cover the tube, and shake the contents. Observe the color of the permanganate solution over a period of five minutes with occasional shaking. Repeat this test with the other two isomeric alcohols.

PROCESSING THE DATA

- 1) Examine models of the various hydrocarbons you tested. Which contain double bonds? Which of the models are planar, which non-planar? Is there an alternate structure for cyclohexane?
- 2)
 - (a) Which of the hydrocarbons were readily oxidized by the alkaline solution of KMnO_4 ?
 - (b) Which was reactive with the bromine solution?
 - (c) What is the relation between the reactivity noted in (a) and (b) and the structure of the hydrocarbons?
- 3) Write the balanced equation for the reaction in which methanol was oxidized by the hot copper oxide.
- 4) What differences were noted when $\text{C}_2\text{H}_5\text{OH}$ reduced KMnO_4 in neutral, acidic, and basic solutions? Assuming that $\text{C}_2\text{H}_5\text{OH}$ was oxidized to acetic acid in each case, write balanced equations for each reaction. Be sure to use the reduction half-reaction which involves the reduction product of manganese which you observed.
- 5) In the reactions involving the three isomeric alcohols with the formula $\text{C}_4\text{H}_9\text{OH}$, what did each of the following tests show about the activity of the functional group $-\text{OH}$ and its position in each alcohol?
 - (a) The test with metallic sodium.
 - (b) The test with concentrated hydrochloric acid.
 - (c) The test with neutral potassium permanganate.
- 6) Write a balanced equation for each case in Question 5(a), (b), and (c) where a reaction occurred.
- 7) *Optional:* There is a fourth alcohol with the formula $\text{C}_4\text{H}_9\text{OH}$. Draw a structural formula for it and name it. How would you predict that it would react with
 - (a) metallic sodium;
 - (b) 12 *M* HCl ;
 - (c) neutral 0.01 *M* KMnO_4 ?

Experiment 40

The Preparation of Esters

If the ionizable hydrogen of an organic acid is replaced by an alkyl group, the compound formed is called an ester. They have pleasant odors and are frequently found in plants and fruits. A general equation showing the formation of an ester is

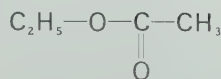


The R and R' are alkyl groups such as $-\text{CH}_3$, $-\text{C}_2\text{H}_5$, and $-\text{C}_3\text{H}_7$. R and R' may be the same or different. Esters, like salts, are named after the compounds from which they are formed. The first part of the name of an ester comes from the alcohol, the second from the acid. The ester *propyl formate* is formed from *propyl* alcohol, and *formic* acid. The salt *sodium nitrate* is formed from the base *sodium* hydroxide and *nitric* acid.

In this experiment, you will prepare ethyl acetate and methyl salicylate.

PROCEDURE

Part I. Preparation of Ethyl Acetate,



- a. Use a 25×200 mm test tube fitted with a one-hole stopper in which is inserted (barely through the stopper) a 60 cm length of 10 mm glass tubing to serve as a reflux condenser.* See Figure 40-1. The tubing must be open at both ends. See Appendix 8, page 139, for glass working techniques.
- b. Place 5 ml of ethanol, 6 ml of 18 M acetic acid, 8–10 drops of 18 M H_2SO_4 , and a boiling chip into the test tube.
- c. **Caution:** Do not heat the reaction mixture directly with a flame since the organic liquids and their vapors are flammable. Clamp the container in an upright position partially immersed in a 250 ml beaker about half full of water. Attach the stopper and reflux condenser tube. Heat the water until the reaction mixture is gently boiling. Continue heating for about 15 minutes. Allow to cool. Note the characteristic odor of the ester in the test tube.

* The reflux condenser is needed for two reasons. First, this reaction proceeds rather slowly. A high temperature gives a practical rate. Second, the components are volatile. The high temperature changes them into gases which escape. The reflux condenser condenses and returns them to the reaction vessel.

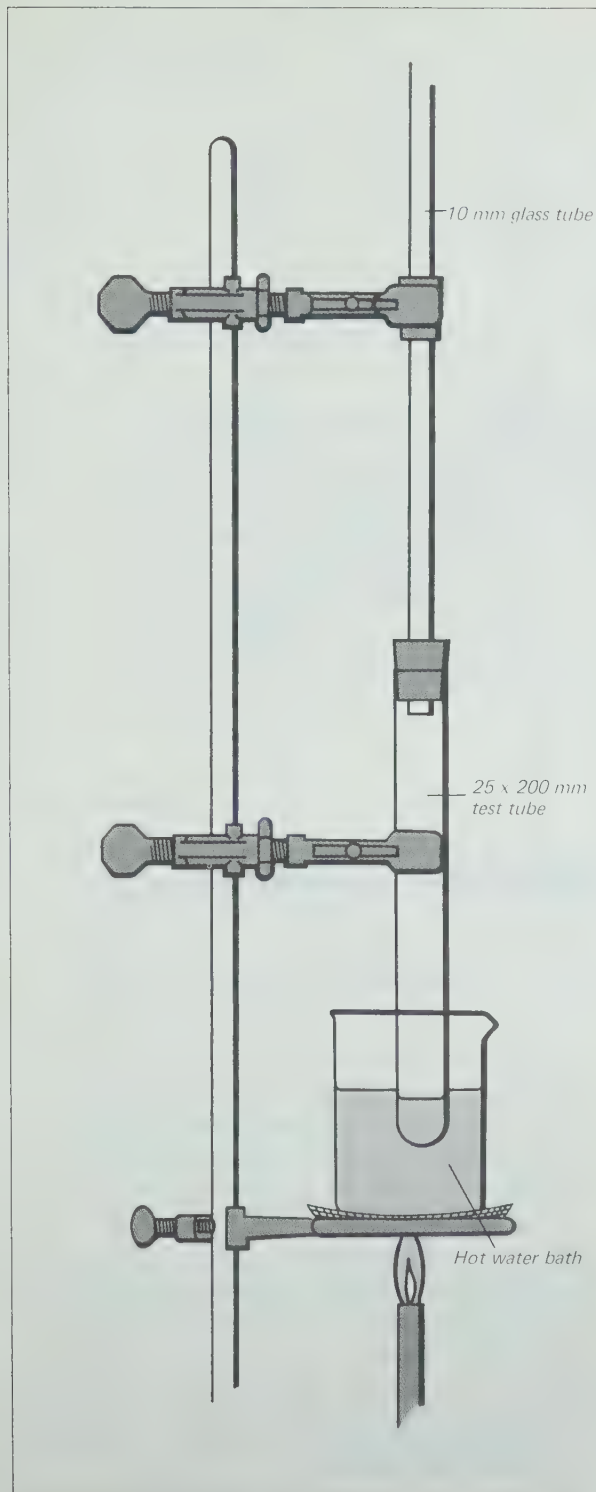


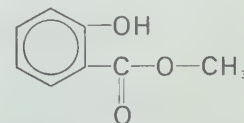
Figure 40-1

Test tube fitted with reflux condenser.

Optional: Purification of the Ester

- d. Attach the test tube to a distilling condenser and heat with a boiling water bath until no more distillate comes over. See Figure 40-2. Collect 10 drops of distillate in each of several 13×100 mm test tubes. Keep the tubes in the order in which distillate is collected in them. What remains behind in the reaction vessel?
- e. Combine the distillates from the tubes that have the most pleasant (fruity) odor. Record the numbers of the tubes used, and the relative intensity of the fruity odor. To this combined distillate, add a few drops of a saturated solution of sodium carbonate. Add sodium carbonate solution until no further visible reaction is evident. Which of the two layers is the water layer?
- f. Separate the two layers by decantation or use a separatory funnel if available. Discard the aqueous solution layer. Dry the ester with 0.5 g of anhydrous calcium chloride (or anhydrous magnesium sulfate) and distill it again if you wish to purify the sample further. Determine the mass of ester produced.

Part II. Preparation of Methyl Salicylate,



- g. Place 3 grams salicylic acid, 10 ml methanol, 20 drops 18 M H_2SO_4 , and a boiling chip into a 25×200 mm test tube.
- h. Attach a reflux condenser as in Part I, step a.
- i. Heat the water bath until the solution in the tube starts to bubble gently and slowly. Keep the water bath at this temperature, about $75^\circ C$, for 15 minutes.
- j. When cool, note the odor of the solution in the tube.

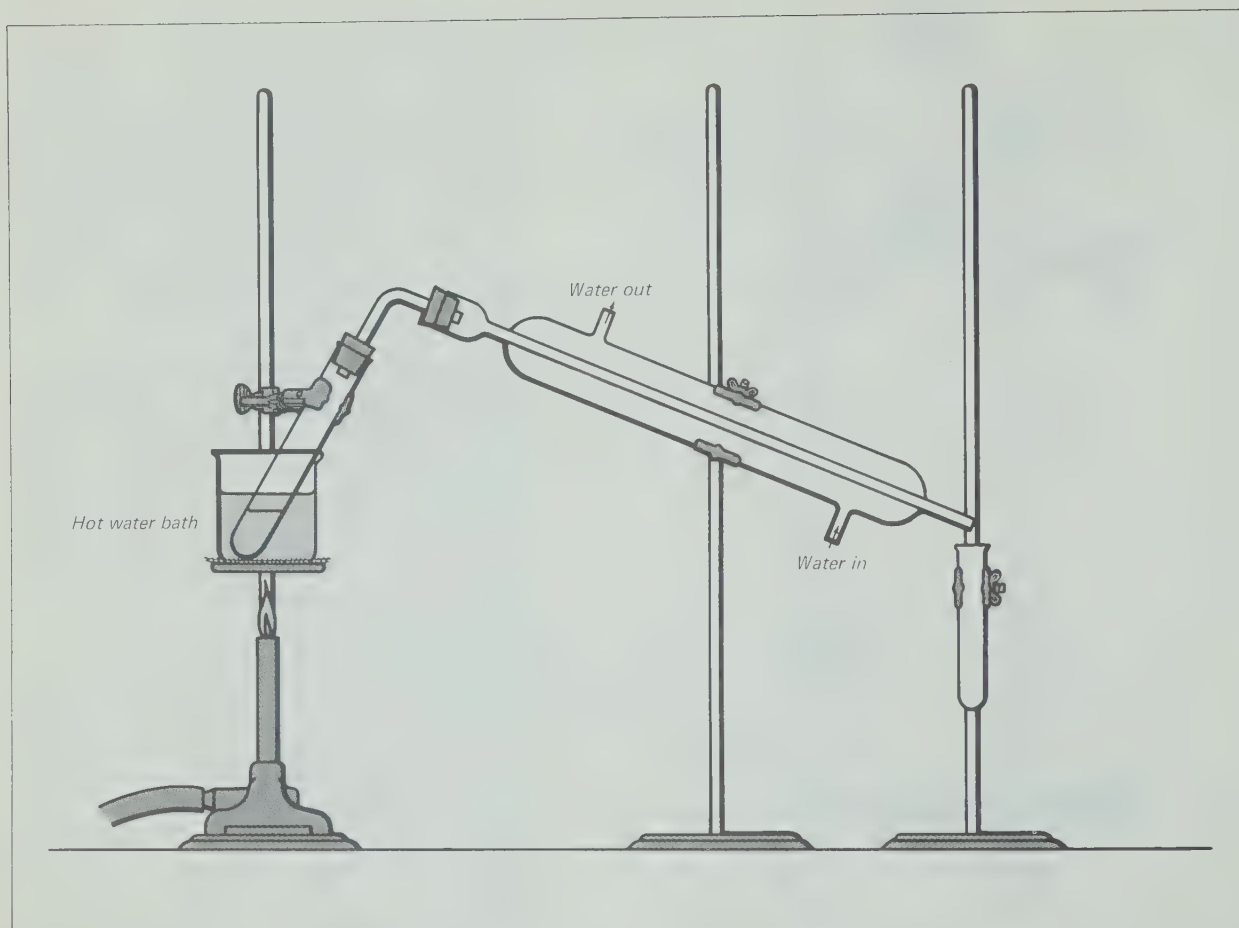


Figure 40-2

Test tube fitted with distilling condenser.

PROCESSING THE DATA

- 1) (a) Calculate the number of moles of each reactant, $\text{C}_2\text{H}_5\text{OH}$ and CH_3COOH , which was used in the preparation of $\text{CH}_3\text{COOC}_2\text{H}_5$.

Data: 1 ml of $\text{C}_2\text{H}_5\text{OH}$ weighs 0.79 g;
1 ml of CH_3COOH weighs 1.05 g.

- (b) Which reactant is present in excess?
(c) If all of one of the reactants were consumed, how many moles of ethyl ace-

tate could be produced? How many grams of $\text{CH}_3\text{COOC}_2\text{H}_5$ is this?

- (d) What is the role of the sulfuric acid in the reaction?
- 2) Write the equation representing the reaction between salicylic acid and methanol to form the ester.
- 3) *Optional:* Explain the observations made in step e concerning the different odors from the different tubes.

Experiment 41

The Electrolysis of Aqueous Potassium Iodide

You will recall that when water undergoes electrolysis the oxidation occurring at the anode produces oxygen gas while the reduction at the cathode produces hydrogen gas. Electrolysis of some aqueous salt solutions, however, may lead to oxidation or reduction of the ions from the salt — if these ions are easier to oxidize or reduce than water.

In this experiment you will electrolyze an aqueous solution of potassium iodide and then identify the products that are formed at the electrodes.

PROCEDURE

- Set up the electrolysis apparatus as shown in Figure 41-1. Add enough 0.5 *M* potassium iodide solution to fill the U-tube about three fourths full. Make the electrical connections and allow the electrolysis to proceed for approximately 15 minutes.
- Note and make a record of any observable products and color changes which occur at either the anode or the cathode. Note the extent to which the brown color diffuses from the anode side into the cathode side of the U-tube.
- Carefully remove the electrodes. Note the odor of the anode. Using a medicine dropper, draw off about 2 ml of the dark brown liquid from the anode side. Add 1 ml of TTE. Cover and shake the test tube for a few seconds. Allow the more dense trichlorotrifluoroethane to settle and note the color of the two liquid layers.
- Use a medicine dropper to draw off 2 ml of the solution on the cathode side. Add a

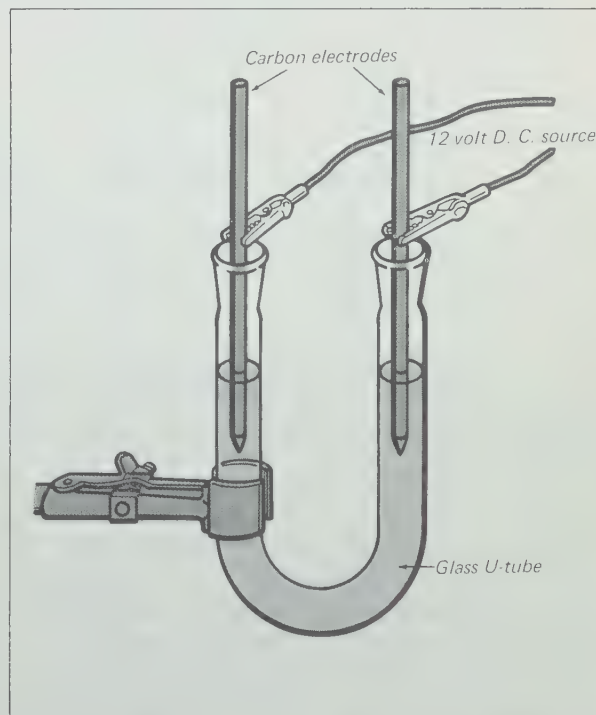


Figure 41-1

An electrolysis apparatus.

few drops of phenolphthalein solution to determine the approximate hydrogen ion concentration of the solution. Add 1 ml of TTE and shake the tube. Then add a few milliliters of 0.1 *M* ferric chloride. Cover and shake the tube.

PROCESSING THE DATA

- 1) Write the equation for the anode half-reaction.
- 2) As iodine is produced at the anode, it reacts with iodide ions to form the complex triiodide ions, I_3^- . These complex ions cause the electrolyte solution to turn brown. Write the equation for this reaction using double arrows to represent equilibrium. What effect did the addition of TTE have on the equilibrium? Use your observations of the changes in color of the two layers as clues to explain the effect.
- 3) Is the solution around the cathode acidic or basic? Write the equation for the cathode half-reaction. Write the equation for the reaction of ferric chloride solution with the sample of solution taken from the cathode side of the U-tube.
- 4) When iodine reacts with a basic solution, it undergoes a self-oxidation-reduction reaction to form iodide ions and iodate ions — both of which are colorless in aqueous solution. Give an explanation of the sharp color boundary noted near the bottom of the U-tube in terms of your knowledge of the products at each electrode. Write the equation for the reaction involved.

Experiment 42

Some Chemistry of Iodine

The following chart contains some of the known compounds of the halogens in various states of oxidation. Note that the range extends from -1 to $+7$ but that fluorine differs significantly from the other halogens in that it has no stable oxyacids.

Making a study of the chart should convince you that oxidation-reduction reactions are a very important part of halogen chemistry.

Although iodine will show some chemistry unique to itself, as every element will, many of its reactions are typical of other halogens. In Experiment 41 you prepared some iodine by electrolysis and observed some reactions which occurred within the U-tube. In this experiment you will investigate these and other reactions of iodine and note the influence of hydrogen ion concentration on the equilibrium reactions.

Oxidation Number	Fluorine	Chlorine	Bromine	Iodine
+7		$\text{HClO}_4, \text{ClO}_4^-$		$\text{HIO}_4, \text{IO}_4^-$
+5		$\text{HClO}_3, \text{ClO}_3^-$	$\text{HBrO}_3, \text{BrO}_3^-$	$\text{HIO}_3, \text{IO}_3^-$
+3		$\text{HClO}_2, \text{ClO}_2^-$		
+1		$\text{HClO}, \text{ClO}^-$	$\text{HBrO}, \text{BrO}^-$	HIO, IO^-
0	F_2	Cl_2	Br_2	I_2
-1	HF, F^-	HCl, Cl^-	HBr, Br^-	HI, I^-

PROCEDURE

Preliminary Experiment — The Starch-Iodine Test

CAUTION: Solid iodine and its vapor will cause burns and stains on skin or clothing. Its vapor is poisonous and even small quantities will irritate the mucous membranes if inhaled. Avoid unnecessary contact.

Prepare a dilute solution of iodine by adding one or two small iodine crystals to about 5 ml of tap water. Warm slightly and add 3 or 4 drops of starch mixture. This is a very sensitive test for molecular iodine. Recall the reaction in Experiment 28.

Part I. Some Reactions of the Iodide Ion, I^-

- Add 2 ml of 0.1 *M* silver nitrate solution to an equal volume of 0.1 *M* potassium iodide solution.
- Add a drop or two of commercial bleach solution, 5% NaOCl , to 2 ml of 0.1 *M* potassium iodide solution mixed with 5 ml of starch mixture. Continue to add the bleach solution until there is a second color change. How do you account for this?
- Add about 5 drops of 3% hydrogen peroxide to a mixture of 2 ml of 0.1 *M* potassium iodide and 5 ml of starch mixture.

Part II. Some Reactions of the Iodate Ion, IO_3^-

- d. Pour about 5 ml of a saturated solution of potassium iodate into each of two test tubes.
- Add 3 ml of 0.1 *M* potassium iodide solution and 2 ml of 6 *M* H_2SO_4 solution to one of the test tubes. Decant. Filter if necessary. Wash the solid with water. Do you recognize the solid? Run an identification test you have used previously to confirm your inference.
 - Add 3 ml of 0.1 *M* potassium iodide solution and 2 ml of 6 *M* potassium hydroxide solution to the second test tube. What effect does changing the hydrogen ion concentration have on the reaction between iodide and iodate ions?
- e. In a dry 18×150 mm test tube, place an amount of solid potassium iodate about the size of a pea and about twice as much sodium metabisulfite, $\text{Na}_2\text{S}_2\text{O}_5$. Clamp the tube in an almost horizontal position and warm it gently with a small flame. Do not heat excessively.

Part III. Reaction of I_2 in a Basic Solution

- f. Use a dropper to add about 10 drops of 6 *M* potassium hydroxide to a few crystals (about 0.5 g) of solid iodine. Shake the test tube gently until the solid iodine disappears and the solution is colorless. You may need to warm the solution gently and add a few more drops of the potassium hydroxide solution. You will identify the product of this reaction in step i.
- g. Cool the solution and make it acidic by adding sufficient 6 *M* nitric acid solution (10 drops or slightly more) to neutralize the base added previously. Note the product of this reaction. What do you think it is?
- h. Make the solution basic again by adding a few drops of 6 *M* potassium hydroxide

solution. Warm gently and add a few more drops of the potassium hydroxide solution if necessary, until a color change is observed. Discard the solution.

- Repeat the procedure outlined in step f. Cool under a cold water tap until a solid crystallizes from the solution. Decant and save both the liquid and the solid.
 - Dry the white solid by heating the test tube gently. Allow it to cool. To the cool white solid add a small amount (about 0.2 g) of solid sodium metabisulfite. Mix the solids by stirring gently with a stirring rod. Heat the mixture with a low flame. Note the result and compare it with that obtained in step e. **Do not inhale the vapors.**
 - To the decanted liquid add 5–10 drops of 0.1 *M* silver nitrate solution. Shake the test tube and note the result. Compare the product to that obtained in step a.

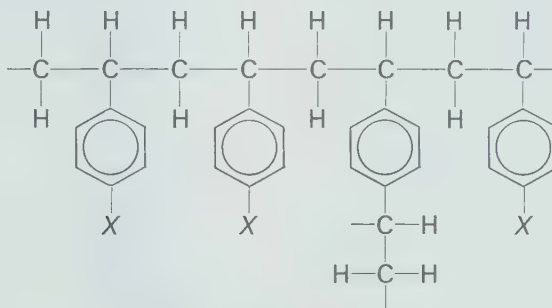
PROCESSING THE DATA

- Write the equations for the reactions observed in steps a, b, and c.
- How did the results in step i(i) compare with those obtained in step e? How did the test with silver nitrate solution in step i(ii) compare with the results of step a? What do you conclude about the ionic species formed when iodine reacts with potassium hydroxide solution as in step f?
- Write the equation for the self-oxidation-reduction reaction of iodine in a basic solution. Write the equation for the reverse of this reaction in acidic solution.

Experiment 43

The Separation of Iron, Cobalt, and Nickel Ions With an Anion Exchange Resin

Ion exchange resins are polymers of high molar mass which contain a framework of covalently bonded carbon and hydrogen atoms. The resins have centers of high positive or negative charge attached to the framework. A typical structure for a portion of a resin polymer is



About one ring in ten is cross-linked into two other chains of the polymer to give it a three-dimensional structure. The groups designated by an $-X$ determine the resin type. If they are $-\text{COOH}$ or $-\text{SO}_3\text{H}$ groups, cations will be exchanged for the hydrogens. This is the type that is used in water softeners in which objectionable ions such as Ca^{2+} and Mg^{2+} are exchanged for H^+ .

If the $-X$ groups are substituted ammonium groups such as $-\text{N}(\text{CH}_3)_3\text{Cl}$, anions will be exchanged for the chloride ions. In this experiment you will use an anion-exchange resin of this type because some of the metal ions used will be converted to complex chloride ions such as FeCl_6^{3-} and $\text{CoCl}_4(\text{H}_2\text{O})_2^-$. For example, when a salt containing the hydrated ferric ion is dissolved in 9 *M* HCl , a complex chloride ion is formed:



The complex anions will be exchanged for the chloride ions of the resin. If the concentration of chloride ions is decreased by using 5 *M* or 1 *M* HCl , some of the complex chloride ions will be changed to form hydrated metal cations which are no longer retained by the resin and can be washed through (eluted from) the column. The chloride complex which forms the least stable complex will

be eluted even at the higher concentration of the chloride ion (9 *M* HCl), while the most stable complex will not be eluted until the chloride ion concentration has been reduced to that of 1 *M* HCl.

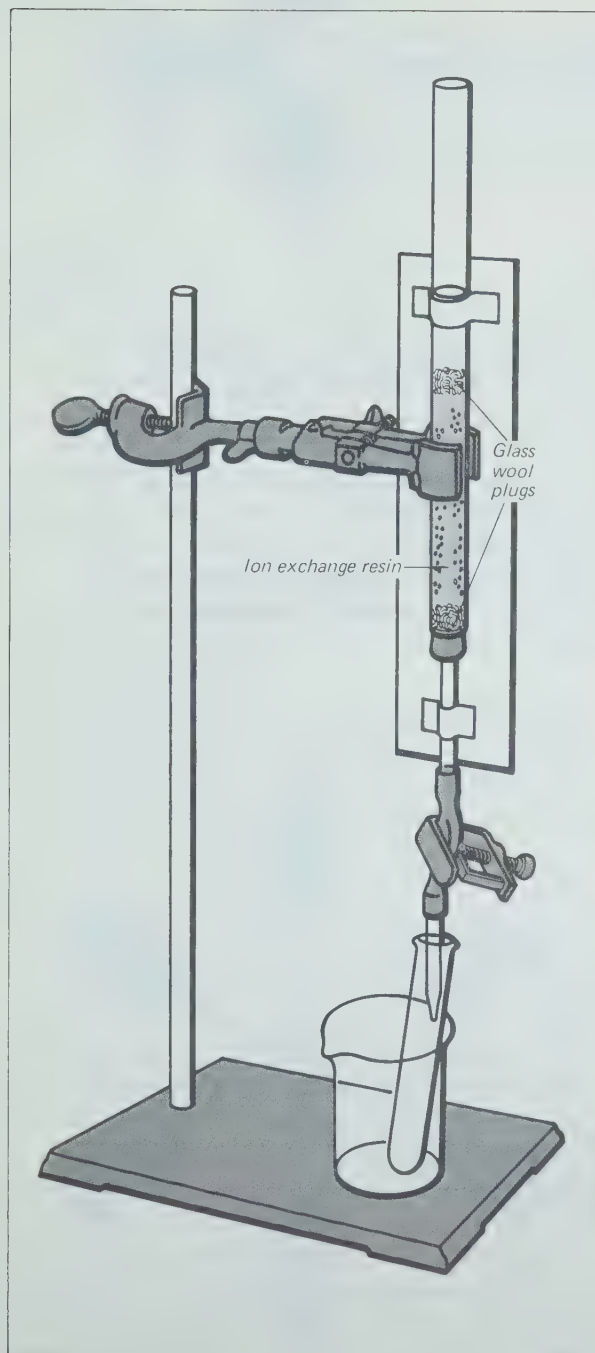
The ions of three closely related metals — iron, cobalt, and nickel — were selected because each has a characteristic color in aqueous solutions containing chloride ions and can thus be readily distinguished as they are eluted from the resin. The test solution you will use contains salts of iron, cobalt, and nickel dissolved in 12 *M* HCl.

After the ions have been separated on the anion exchange column you will test the eluted samples to determine the efficiency of the separations.

PROCEDURE

- a. Obtain an ion exchange column and set it up as shown in Figure 43-1. Attach a piece of white paper to the column. This will provide a good background against which the colors of the solutions can be readily seen. The solution on the column is 2 *M* HCl. Leave this on the column until you are ready to add another solution. *Do not allow the column to run dry* since this may lead to uneven flow (channeling) later and decrease the efficiency of the separation. *Either add the next solution or close the screw clamp whenever the level of the liquid in the tube drops to the glasswool plug at the top of the column.*
- b. Obtain 50 ml of 9 *M* HCl solution. From this prepare 45 ml of 1 *M* HCl solution by diluting 5 ml of 9*M* HCl with 40 ml of distilled water. Prepare 30 ml of 5 *M* HCl by mixing 15 ml of 9 *M* HCl with 15 ml of 1 *M* HCl. Place each of these solutions in clean labeled beakers for use when needed. Obtain 2 ml of the test solution containing the three ions to be separated.
- c. Open the screw clamp to allow the 2 *M* HCl solution already on the column to drop to the level of the top glasswool plug. Add 9 *M* HCl to refill to the top of the tube. Adjust the screw clamp so the flow will be about 2.5 ml per minute.
- d. When the 9 *M* HCl has dropped to the top of the glasswool plug, close the clamp and *add the 2 ml of the colored test solution.*
- e. When the level again drops to the glasswool plug, add 5 ml of 9 *M* HCl. Collect 5 ml of **eluant** (the solution that passes through the column). Add another 5 ml portion of 9 *M* HCl and collect 5 ml of eluant in a separate test tube. Repeat this procedure with another 5 ml of 9 *M* HCl. Label the three test tubes e-1, e-2, and e-3. Note the color of the eluant in each test tube.
- f. Repeat step e, except use four 5 ml portions of 5 *M* HCl solution in place of the 9 *M* HCl. Collect the eluant, about 5 ml in each of four test tubes labeled f-1, f-2, f-3, and f-4.
- g. Repeat step e, using four or five 5 ml portions of 1 *M* HCl solution. Collect the eluant, about 5 ml in each test tube labeled g-1, g-2, etc.
- h. After the last 5 ml portion of 1 *M* HCl solution drops to the glasswool plug at the top of the column, add 10 ml of distilled water to rinse the resin. Dilute 4 ml of 9 *M* HCl with 14 ml of water to make some 2 *M* HCl solution. Add this to the column so it will be ready for use again by another student. Close the clamp when the acid level is about 2 cm above the top of the glasswool plug.
- i. Identify the different colored solutions by comparing them with hydrochloric acid solutions of iron, cobalt, and nickel chlorides which your teacher will have available.
- j. Test a drop of each eluted sample separately with each of the following reagents to determine the efficiency of the separation.

Open the clamp to obtain the same rate of flow as before.

**Figure 43-1**

An ion-exchange column.

Place the drops on a piece of plastic film which is covering a white paper

- i) Add a drop of 0.1 *M* KSCN solution. A blood-red coloration due to formation of $\text{Fe}(\text{SCN})^{2+}$ ion indicates the presence of Fe^{3+} ion.
 - ii) Place a fresh drop of each eluant on the plastic film. Make each basic by adding a few drops of 15 *M* NH_3 solution. **Caution:** The solution will get hot during neutralization. Use litmus to check when basic, then add a drop of dimethyl glyoxime reagent. A brilliant red precipitate indicates the presence of nickel.
 - iii) Place a fresh drop of each eluant on the plastic film. Add a drop of 10% solution of NH_4SCN in acetone to each drop being tested. The appearance of a blue color due to the formation of $\text{Co}(\text{SCN})_4^{2-}$ ions indicates the presence of Co^{2+} ions.
- Record all observations neatly, in tabular form when appropriate.

PROCESSING THE DATA

- 1) One of the metallic ions was eluted with 9 *M* HCl. It would also have been eluted with the 5 *M* or 1 *M* HCl solutions. Which one was it? Give a reason for this ease of elution in terms of the stability of its complex chloride anions.
- 2) The color of some of the ions on the column was different from the color of the final eluant. Explain this in terms of the composition of the complex ions.
- 3) What are the conditions which favor the release of ions from the column?
- 4) Recalling that the resin used is an anion exchange type, which of the metallic ions in this experiment forms the most stable complex chloride anion?

Experiment 44

Preparation of a Complex Salt

Many salts crystallize out of aqueous solution as hydrates—for example, $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, or $\text{Al}_2(\text{SO}_4)_3 \cdot 9 \text{H}_2\text{O}$. The building blocks in the crystals are hydrated cations and anions such as $\text{Cu}(\text{H}_2\text{O})_4^{2+}$, and $\text{SO}_4(\text{H}_2\text{O})_2^{2-}$ in the case of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$.

Many metals form stable complex ions in which the metal ion is bound to molecules (or ions) other than water—for example, $\text{Co}(\text{NH}_3)_6^{3+}$ or $\text{Fe}(\text{CN})_6^{3-}$. Salts containing such complex ions as these are called coordination compounds or complex salts—for example, hexaamminecobalt(III) chloride, $\text{Co}(\text{NH}_3)_6\text{Cl}_3$, and potassium hexacyanoferrate(III), $\text{K}_3\text{Fe}(\text{CN})_6$. The complex ions found in these crystalline solids are shown in Figure 44-1.

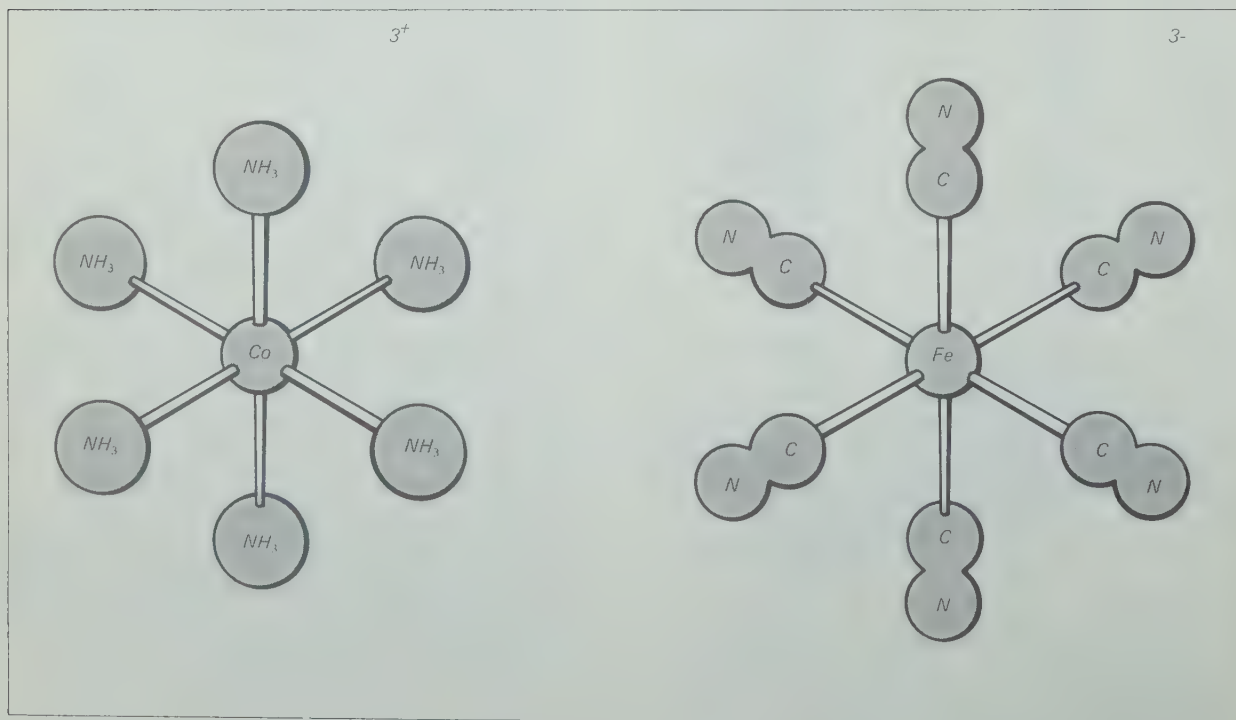


Figure 44-1

The structure of some complex ions.

Naming complex salts emphasizes a current problem with nomenclature. An old system is in the process of being replaced by a new, more informative system: the Stock system. There are two names for CuCl_2 . It is called cupric chloride in the old system, and copper(II) chloride in the Stock system. Since both systems are still in use, both are used in this experiment.

PROCEDURE

Part I. Preparation of a Complex Salt: Tetraamminecopper(II) Sulfate Monohydrate, $\text{Cu}(\text{NH}_3)_4\text{SO}_4 \cdot \text{H}_2\text{O}$

- Dilute 8 ml of 15 M $\text{NH}_3(\text{aq})$ with 5 ml of distilled water in a small beaker.
- Obtain 0.02 mole of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (249.5 g/mole). Use a mortar and pestle to pulverize the cupric sulfate crystals. Add the powdered crystals to the ammonia solution and stir until all of the cupric sulfate pentahydrate is dissolved.
- Pour 8 ml of ethyl alcohol slowly down the side of the beaker so as to cover the solution with alcohol. Do not stir or agitate the mixture. Cover with a watch glass and let it stand overnight.
- After the mixture has been allowed to stand overnight, stir slowly and gently to insure complete precipitation. Allow the crystals to settle. Decant and discard the liquid. Transfer the crystals to a filter by rinsing with 3–5 ml portions of a mixture of equal volumes of 15 M $\text{NH}_3(\text{aq})$ and ethyl alcohol.
- Finally, wash the crystals on the funnel with 5 ml of ethyl alcohol. Attach the hose from an aspirator to the funnel stem to draw the liquid from the crystals. Apply the suction gently to avoid tearing the filter paper. Repeat the washing and drying operation.
- Remove the filter paper and spread it out on a paper towel.
- Weigh the dry crystals. Did you obtain 0.02 mole of the complex salt? How many moles of NH_3 were used?

Part II. Reactions of Complex Ions

- Place 1 ml of *anhydrous* cupric sulfate crystals into a small, dry test tube. Note the change in color which takes place when 2 or 3 ml of water are added. Now add 6 M ammonia solution a few drops at a time until 5 ml have been added. Record your observations.
- Dissolve a small amount of the complex salt you prepared in Part I in 5 ml of water in a small test tube. What ionic species present in the solution are responsible for the color? Add the solution to 20 ml of water in a beaker, and record any color changes.
- Place a small quantity of the dry salt you prepared in Part I into a small test tube. Heat gently and note any color changes. Identify the gas given off.
- Place 1 ml of *hydrated* cupric sulfate into a small test tube. Heat gently and note any color changes. Identify the gas given off.

PROCESSING THE DATA

- In Part II h, account for the successive color changes noted in terms of the structure of the ion containing copper.
- What are the ionic species present when the complex salt tetraamminecopper(II) sulfate is dissolved in a small quantity of water? Give a reasonable explanation of the changes which took place as more water was added.
- Account for the changes you noted when each salt was heated in Parts II j and k.
- Why was a yield of 0.02 mole expected for the salt you prepared in Part I?

Experiment 45

Preparation of Potassium Dichromate

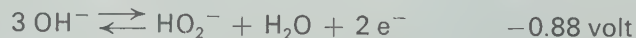
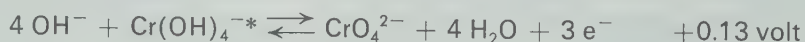
The transition metals can form compounds containing the elements in a variety of oxidation states. In this experiment and in Experiment 46 you will study some of the oxidation-reduction chemistry of chromium. You will prepare some of the common compounds of this element. In the chart below note the oxidation number of chromium, the formulas, and the color for the ionic species present in acidic and basic solutions. Use this chart to follow the reactions as you prepare potassium dichromate from hydrated chromium(III) acetate, $\text{Cr}(\text{CH}_3\text{COO})_3 \cdot \text{H}_2\text{O}$.

Oxidation Number of Chromium	Ionic Species Present	
	Acidic Solution	Basic Solution
+6	$\text{Cr}_2\text{O}_7^{2-}$ (orange)	CrO_4^{2-} (yellow)
+3	$\text{Cr}(\text{H}_2\text{O})_6^{3+}$ (violet)	$\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$ (green)

Hydrogen peroxide is a suitable oxidizing agent in a basic solution. Since H_2O_2 is a weak acid, some HO_2^- ions are present in the solution.

Oxidation Number of Oxygen	Molecular or Ionic Species Present	
	Acidic Solution	Basic Solution
-1	H_2O_2	HO_2^-
-2	H_2O or H_3O^+	OH^-

The E° values and the equations for the half-reactions in basic solution are:



Before you proceed with the preparation, balance the equations for the reactions involved so that you can calculate the number of moles of reactants required for each of the steps in the preparation. Record these in your laboratory notebook before coming to the laboratory.

Reaction 1. Conversion of $\text{Cr}(\text{H}_2\text{O})_6^{3+}$ to $\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$ with an excess of strong base.

Reaction 2. Oxidation of $\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$ to CrO_4^{2-} by peroxide in a basic solution.

Reaction 3. Conversion of CrO_4^{2-} to $\text{Cr}_2\text{O}_7^{2-}$ by acidification with CH_3COOH .

* $\text{Cr}(\text{OH})_4^-$ is a simplified formula for the ion $\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$ without the water of hydration.

PROCEDURE

- Weigh out 0.01 mole of chromium(III) acetate (247.2 g/mole). Dissolve it in about 20 ml of distilled water in a small beaker. Warm the solution to dissolve the solid.
- Pour 25 ml of 6 *M* potassium hydroxide solution into a 250 ml conical flask. Add the chromium(III) acetate solution slowly to the flask. Swirl the flask to mix the solutions and to make sure that any chromium(III) hydroxide formed will dissolve in the excess base present.
- Refer to the equation you balanced for *Reaction 2* and note the number of moles of hydrogen peroxide required to oxidize the 0.01 mole of chromium(III) ion. Calculate the volume of commercial hydrogen peroxide, 3% by mass, required to obtain this many moles. Add about three times the calculated amount of hydrogen peroxide solution. Add 1 or 2 boiling chips, and heat the flask to boiling. If the solution does not become yellow in color after boiling for a minute or two, add about 10 ml more hydrogen peroxide solution and bring to a boil again. Repeat if necessary but avoid using more H_2O_2 than is needed.
- After the solution turns yellow, boil it gently until its volume has been reduced to about 30 milliliters. Any excess hydrogen peroxide should be decomposed during the boiling.
- Allow the solution to cool for a few minutes. Acidify the solution by adding concentrated acetic acid, 18 *M* CH_3COOH , drop by drop until the solution becomes orange in color. The color of the dichromate ion in solution is orange. Add 1 ml of 18 *M* acetic acid in excess.
- Heat the solution to boiling and evaporate the solution to a volume of about 25 ml. Cool the solution using an ice bath to crystallize the potassium dichromate.
- Filter the solid and wash with two 5 ml portions of ethyl alcohol. Dry and weigh the crystals. Compare the quantity obtained

with the theoretical amount expected, as calculated from the equations.

PROCESSING THE DATA

- Write the equation for each of the reactions involved in the preparation of potassium dichromate. Show your calculations for the amount of hydrogen peroxide required and for the expected yield of potassium dichromate.
- What ionic species were present in the final acidic solution before it was cooled? What equilibria are established?
- Considering the following solubility data, would you expect your sample of potassium dichromate to be relatively pure? Why?

Temperature, °C	Solubility in g/100 g of water	
	KCH_3COO	$\text{K}_2\text{Cr}_2\text{O}_7$
0	217	5
20	256	12
40	323	26
60	350	43
80	380	61
100	—	80

- Compare the E° values of the half-reactions for the oxidation of Cr(III) to Cr(VI) in a basic solution with that for the oxidation in an acidic solution. See the introductory section of this experiment for the former value and the Appendix 4 for the latter value. Predict which oxidation will favor products more at equilibrium.
- Note the half-reaction for the oxidation of H_2O_2 for which the E° value is -0.68 volt (Appendix 4). Can you now suggest a reason for decomposing the excess H_2O_2 in step d before the solution was acidified to convert chromate ions to dichromate ions?
- Is the conversion of chromate ion to dichromate ion an oxidation reaction? Explain.



Experiment 46

Preparation of Chrome Alum

In Experiment 45 you prepared potassium dichromate from chromium(III) acetate by oxidation in a basic solution. In this experiment you will reverse the process and reduce potassium dichromate with sulfur dioxide in an acidic solution to produce a chromium(III) salt called chrome alum. Study the chart listing various oxidation numbers for chromium given in Experiment 45 and note the changes in color and ionic species which will be involved.

Refer to Appendix 4 for the E° values for the reduction half-reaction of Cr(VI) to Cr(III) and the oxidation half-reaction of SO_2 to SO_4^{2-} , both in acid solution. Before coming to the laboratory, balance the equation for this oxidation-reduction reaction and record it in your laboratory notebook.

PROCEDURE

- Weigh out 0.05 mole of $\text{K}_2\text{Cr}_2\text{O}_7$ (294.2 g/mole) and add it to 60 ml of 3 *M* H_2SO_4 . Warm to dissolve the potassium dichromate. Set aside to cool to room temperature.
- Set up the sulfur dioxide generator shown in Figure 46-1. Clamp the flask on an iron ring over a wire screen so that a burner may be used to heat the flask if necessary. How many moles of SO_2 are required to reduce 0.05 mole of potassium dichromate? See the equation you balanced previously. How many moles of $\text{Na}_2\text{S}_2\text{O}_5$ should be used to produce this sulfur dioxide by reaction with 6 *M* sulfuric acid? Convert the number of moles to grams of $\text{Na}_2\text{S}_2\text{O}_5$ and use about double this amount in the generator.
- Add about 50 ml of tap water to the solid $\text{Na}_2\text{S}_2\text{O}_5$ in the generator. Attach a 10 cm piece of glass tubing to the hose leading from the generator and place it in the 125 ml conical flask.
- Cool the potassium dichromate solution to about 25°C before starting to generate the SO_2 . **Caution:** Use an ice bath when necessary to maintain the potassium dichromate solution below 40°C during the reduction of the dichromate. This is necessary to prevent complicating side reactions. Use a fume hood if available.
- Add 10 ml of 6 *M* sulfuric acid, a few ml at a time, to the generator to produce a steady flow of SO_2 gas. If the reaction slows, add another 10 ml, a few ml at a time. After a total of 20 ml of acid has been added, warm the generator with a low flame from time to time as necessary.
- Allow the reaction to continue until the solution turns from orange to a dark blue-green color. Stir or swirl the solution periodically. Keep the temperature below 40° Celsius. To check the color of the solution, place a drop on a filter paper and note the circle of color produced as the solution diffuses. The reduction is complete when

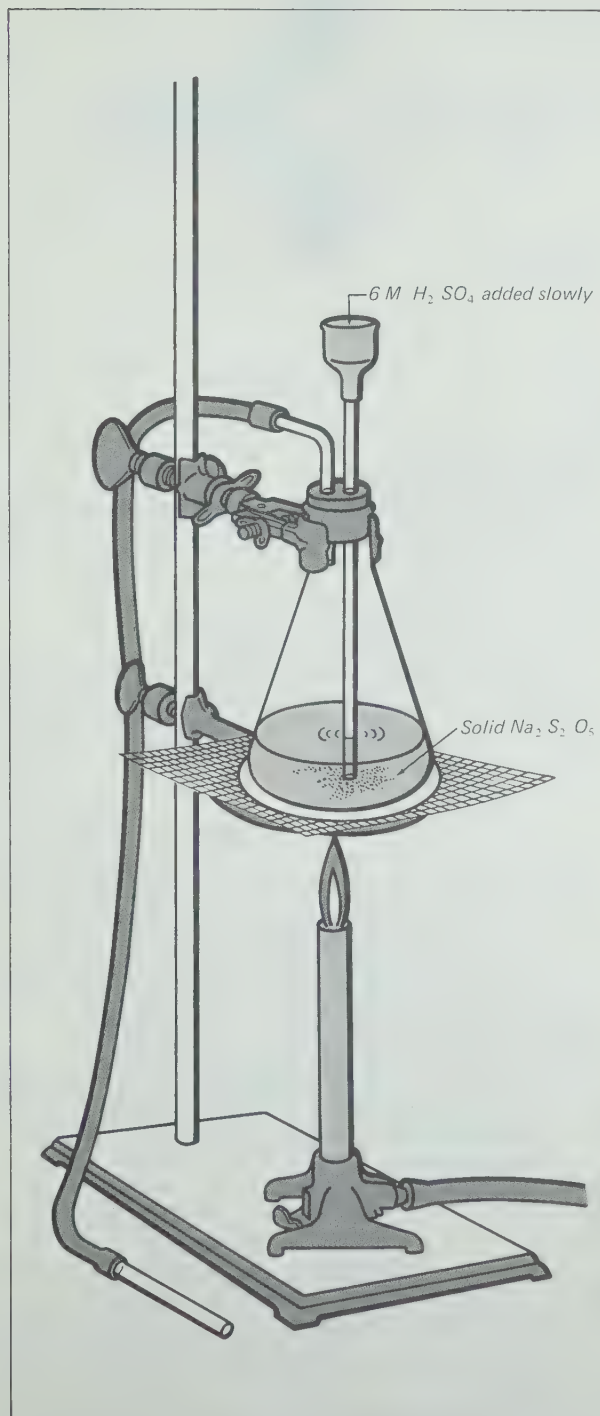


Figure 46-1

A sulfur dioxide generator.

no orange color is noted before the spot dries. The estimated time for the reaction is about 30 minutes.

- g. Pour the solution into an evaporating dish. Cover loosely and allow it to stand until crystals of chrome alum have formed. If the crystallization is allowed to proceed slowly, over a period of several days, larger crystals will be formed. Filter and allow to dry.
- h. Weigh the dry crystals and calculate the percent yield you obtained.

PROCESSING THE DATA

- 1) Under what conditions could you grow large crystals of chrome alum?
- 2) Examine one of the larger crystals you prepared. Note any outstanding features of its geometric symmetry and prepare a sketch showing them.
- 3) What are the building blocks of the chrome alum crystal? Draw a three-dimensional structural formula for each of the ionic species involved.
- 4) Show calculations of the amounts of reactants required and the theoretical yield expected.

Appendix 1

Measurement — The Metric System

The metric system of measurement has several advantages over our more familiar English system. It has, therefore, been widely accepted as *the* system of measurement used by scientists throughout the world. The advantages of this system will become obvious to you as you use it in working the exercises that follow, and as you make measurements and calculations throughout this course.

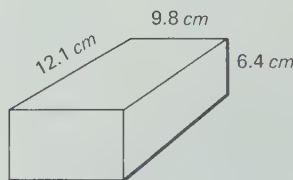
Length. In the metric system, the **meter** is the standard unit of length. It is a bit longer than one yard. The meter is divided into 10 pieces, each of which is a decimeter. The decimeter is divided into 10 pieces, each of which is a centimeter. The meter contains 100 centimeters. The centimeter is divided into 10 pieces, each of which is a millimeter. The meter contains 1000 millimeters.

Mass: The standard unit of mass is the **kilogram**. One kilogram contains 1000 grams. A gram is a relatively small unit of mass. A five-cent coin has a mass of 5 grams. There are about 454 grams per pound of mass. See Figure 3-1, page 4.

Volume. The standard unit of volume is the **liter**. A liter is a little larger than a quart. A liter is the volume of a cube 10 centimeters on each edge. One liter of water at 4°C has a mass of 1000 grams. A liter is divided into 1000 parts, each of which is called a milliliter. A milliliter is the volume of a cube 1 cm on each edge. Thus 1 ml = 1 cm³ (sometimes written 1 cc). See Figure 3-2, page 5.

EXERCISES

Do the following exercises, using the dimensions of a block as shown.



length = 12.1 cm
width = 9.8 cm
height = 6.4 cm

- 1) Add two times the length to two times the width to obtain the perimeter of the largest face. Express your answer to the nearest 0.1 cm. Also express your answer in meters.
- 2) Calculate the area of the largest face.
- 3) Determine the volume of the block.
If the block had been measured in English units, its dimensions could have been recorded as:

length = $4\frac{3}{4}$ inches
width = $3\frac{7}{8}$ inches
height = $2\frac{1}{2}$ inches

- 4) Find the perimeter of the largest face. Express your answer in both inches and in feet.
- 5) Calculate the area of the largest face.
- 6) Find the volume of the block using English units.

SUMMARY OF METRIC UNITS

Measure	Standard Unit	Related Units
Length	meter	1 kilometer = 1000 meters 1 decimeter = 0.1 meter 1 centimeter = 0.01 meter 1 millimeter = 0.001 meter
Mass	kilogram	1 kilogram = 1000 grams 1 decigram = 0.1 gram 1 centigram = 0.01 gram 1 milligram = 0.001 gram
Volume	liter	1 deciliter = 0.1 liter 1 centiliter = 0.01 liter 1 milliliter = 0.001 liter
<i>Note:</i> kilo = 1000 deci = 0.1		centi = 0.01 milli = 0.001

METRIC-ENGLISH EQUIVALENT UNITS

1 kilogram = 2.20 pounds	1 pound = 454 grams
1 meter = 39.37 inches	1 inch = 2.54 centimeters
1 liter = 1.06 U.S. fluid quarts	1 ounce, U.S. fluid = 30 milliliters
	1 ounce, U.S. (avdp.) = 28 grams
	1 ounce, U.S. (troy) = 31 grams

ADDITIONAL EXERCISES

- 7) Change:
- 454 grams to kilograms
 - 3.2 kilograms to grams to milligrams
 - 13 millimeters to centimeters to meters
 - 760 millimeters to centimeters to meters
 - 4.1 meters to centimeters to millimeters
 - 1500 milliliters to liters
 - 250 milliliters to liters
 - 1.3 liters to milliliters
- 8) Calculate the mass of 323 cm³ of water at 4°C.
- 9) Change:
- 2 pounds to ounces
 - 2 tons to pounds
 - 2 feet to inches
 - 2000 feet to miles

- 119 cm²
- 760 cm³
- 17¹/₄ inches, 1.44 feet
- 18¹³/₃₂ inches²
- 46¹/₆₄ inches³
- 0.454 kg
 - 3200 g, 3,200,000 mg
 - 1.3 cm, 0.013 m
 - 76 cm, 0.76 m
 - 410 cm, 4100 mm
 - 1.5 liters
 - 0.25 liter
 - 1300 ml
- $323 \text{ cm}^3 \times \frac{1 \text{ gram}}{\text{cm}^3} = 323 \text{ grams}$
- 32 ounces
 - 4000 pounds
 - 24 inches
 - 0.379 mile

ANSWERS TO EXERCISES

- 1) 43.8 cm, 0.438 m

Appendix 2

NAMES, FORMULAS, AND CHARGES OF SOME COMMON IONS

POSITIVE IONS (CATIONS)		NEGATIVE IONS (ANIONS)	
aluminum	Al^{3+}	acetate	CH_3COO^-
ammonium	NH_4^+	bromide	Br^-
barium	Ba^{2+}	carbonate	CO_3^{2-}
calcium	Ca^{2+}	hydrogen carbonate ion, bicarbonate	HCO_3^-
chromium(II), chromous	Cr^{2+}	chlorate	ClO_3^-
chromium(III), chromic	Cr^{3+}	chloride	Cl^-
copper(I),* cuprous	Cu^+	chlorite	ClO_2^-
copper(II), cupric	Cu^{2+}	chromate	CrO_4^{2-}
hydrogen, hydronium	$\text{H}^+, \text{H}_3\text{O}^+$	dichromate	$\text{Cr}_2\text{O}_7^{2-}$
iron(II),* ferrous	Fe^{2+}	fluoride	F^-
iron(III), ferric	Fe^{3+}	hydroxide	OH^-
lead	Pb^{2+}	hypochlorite	ClO^-
lithium	Li^+	iodide	I^-
magnesium	Mg^{2+}	nitrate	NO_3^-
manganese(II), manganous	Mn^{2+}	nitrite	NO_2^-
mercury(I),* mercurous	Hg_2^{2+}	oxalate	$\text{C}_2\text{O}_4^{2-}$
nickel	Ni^{2+}	hydrogen oxalate ion, binoxalate	HC_2O_4^-
potassium	K^+	perchlorate	ClO_4^-
silver	Ag^+	permanganate	MnO_4^-
sodium	Na^+	phosphate	PO_4^{3-}
tin(II),* stannous	Sn^{2+}	monohydrogen phosphate	HPO_4^{2-}
tin(IV), stannic	Sn^{4+}	dihydrogen phosphate	H_2PO_4^-
zinc	Zn^{2+}	sulfate	SO_4^{2-}
		hydrogen sulfate ion, bisulfate	HSO_4^-
		sulfide	S^{2-}
		hydrogen sulfide ion, bisulfide	HS^-
		sulfite	SO_3^{2-}
		hydrogen sulfite ion, bisulfite	HSO_3^-

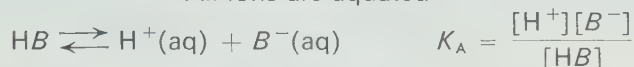
* Aqueous solutions are readily oxidized by air.

Note: In ionic compounds the relative number of positive and negative ions is such that the sum of their electric charges is zero.

Appendix 3

RELATIVE STRENGTHS OF ACIDS IN AQUEOUS SOLUTION AT ROOM TEMPERATURE

All ions are aquated



ACID	STRENGTH	REACTION	K_A
perchloric acid	very strong	$HClO_4 \rightarrow H^+ + ClO_4^-$	very large
hydroiodic acid		$HI \rightarrow H^+ + I^-$	very large
hydrobromic acid		$HBr \rightarrow H^+ + Br^-$	very large
hydrochloric acid		$HCl \rightarrow H^+ + Cl^-$	very large
nitric acid		$HNO_3 \rightarrow H^+ + NO_3^-$	very large
sulfuric acid	strong	$H_2SO_4 \rightarrow H^+ + HSO_4^-$	large
hydrated hydrogen ion		$H_3O^+ \rightarrow H^+ + H_2O$	1.0
oxalic acid		$HOCCOOH \rightarrow H^+ + HOCCOO^-$	5.4×10^{-2}
sulfurous acid ($SO_2 + H_2O$)		$H_2SO_3 \rightarrow H^+ + HSO_3^-$	1.7×10^{-2}
hydrogen sulfate ion		$HSO_4^- \rightarrow H^+ + SO_4^{2-}$	1.3×10^{-2}
phosphoric acid	weak	$H_3PO_4 \rightarrow H^+ + H_2PO_4^-$	7.1×10^{-3}
ferric ion		$Fe(H_2O)_6^{3+} \rightarrow H^+ + Fe(H_2O)_5(OH)^{2+}$	6×10^{-3}
hydrogen telluride		$H_2Te \rightarrow H^+ + HTe^-$	2.3×10^{-3}
hydrofluoric acid		$HF \rightarrow H^+ + F^-$	6.7×10^{-4}
nitrous acid		$HNO_2 \rightarrow H^+ + NO_2^-$	5.1×10^{-4}
hydrogen selenide	weak	$H_2Se \rightarrow H^+ + HSe^-$	1.7×10^{-4}
chromic ion		$Cr(H_2O)_6^{3+} \rightarrow H^+ + Cr(H_2O)_5(OH)^{2+}$	10^{-4}
benzoic acid		$C_6H_5COOH \rightarrow H^+ + C_6H_5COO^-$	6.6×10^{-5}
hydrogen oxalate ion		$HOCCOO^- \rightarrow H^+ + OOC-COO^{2-}$	5.4×10^{-5}
acetic acid		$CH_3COOH \rightarrow H^+ + CH_3COO^-$	1.8×10^{-5}
aluminum ion	weak	$Al(H_2O)_6^{3+} \rightarrow H^+ + Al(H_2O)_5(OH)^{2+}$	10^{-5}
carbonic acid ($CO_2 + H_2O$)		$H_2CO_3 \rightarrow H^+ + HCO_3^-$	4.4×10^{-7}
hydrogen sulfide		$H_2S \rightarrow H^+ + HS^-$	1.0×10^{-7}
dihydrogen phosphate ion		$H_2PO_4^- \rightarrow H^+ + HPO_4^{2-}$	6.3×10^{-8}
hydrogen sulfite ion		$HSO_3^- \rightarrow H^+ + SO_3^{2-}$	6.2×10^{-8}
ammonium ion	very weak	$NH_4^+ \rightarrow H^+ + NH_3$	5.7×10^{-10}
hydrogen carbonate ion		$HCO_3^- \rightarrow H^+ + CO_3^{2-}$	4.7×10^{-11}
hydrogen telluride ion		$HTe^- \rightarrow H^+ + Te^{2-}$	10^{-11}
hydrogen peroxide		$H_2O_2 \rightarrow H^+ + HO_2^-$	2.4×10^{-12}
monohydrogen phosphate ion		$HPO_4^{2-} \rightarrow H^+ + PO_4^{3-}$	4.4×10^{-13}
hydrogen sulfide ion	very weak	$HS^- \rightarrow H^+ + S^{2-}$	1.3×10^{-13}
water		$H_2O \rightarrow H^+ + OH^-$	$1.8 \times 10^{-16*}$
hydroxide ion		$OH^- \rightarrow H^+ + O^{2-}$	$< 10^{-36}$
ammonia		$NH_3 \rightarrow H^+ + NH_2^-$	very small

* $K_w = K_A(55.5) = 1 \times 10^{-14}$

Appendix 4

STANDARD OXIDATION POTENTIALS FOR HALF-REACTIONS IONIC CONCENTRATIONS, 1 M IN WATER AT 25°

All ions are aquated

HALF-REACTION		E° (volts)	
Very strong reducing agents ↑ Reducing strength increases	$\text{Li} \longrightarrow e^- + \text{Li}^+$	3.00	Very weak oxidizing agents ↓ Oxidizing strength increases
	$\text{Rb} \longrightarrow e^- + \text{Rb}^+$	2.92	
	$\text{K} \longrightarrow e^- + \text{K}^+$	2.92	
	$\text{Cs} \longrightarrow e^- + \text{Cs}^+$	2.92	
	$\text{Ba} \longrightarrow 2e^- + \text{Ba}^{2+}$	2.90	
	$\text{Sr} \longrightarrow 2e^- + \text{Sr}^{2+}$	2.89	
	$\text{Ca} \longrightarrow 2e^- + \text{Ca}^{2+}$	2.87	
	$\text{Na} \longrightarrow e^- + \text{Na}^+$	2.71	
	$\text{Mg} \longrightarrow 2e^- + \text{Mg}^{2+}$	2.37	
	$\text{Al} \longrightarrow 3e^- + \text{Al}^{3+}$	1.66	
	$\text{Mn} \longrightarrow 2e^- + \text{Mn}^{2+}$	1.18	
	$\text{H}_2(\text{g}) + 2\text{OH}^- \longrightarrow 2e^- + 2\text{H}_2\text{O}$	0.83	
	$\text{Zn} \longrightarrow 2e^- + \text{Zn}^{2+}$	0.76	
	$\text{Cr} \longrightarrow 3e^- + \text{Cr}^{3+}$	0.74	
	$\text{H}_2\text{Te} \longrightarrow 2e^- + \frac{1}{8}\text{Te}_8 + 2\text{H}^+$	0.72	
	$2\text{Ag} + \text{S}^{2-} \longrightarrow 2e^- + \text{Ag}_2\text{S}$	0.69	
	$\text{Fe} \longrightarrow 2e^- + \text{Fe}^{2+}$	0.44	
	$\text{H}_2(\text{g}) \longrightarrow 2e^- + 2\text{H}^+ (10^{-7} M)$	0.414	
	$\text{Cr}^{2+} \longrightarrow e^- + \text{Cr}^{3+}$	0.41	
	$\text{H}_2\text{Se} \longrightarrow 2e^- + \frac{1}{8}\text{Se}_8 + 2\text{H}^+$	0.40	
	$\text{Pb} + \text{SO}_4^{2-} \longrightarrow 2e^- + \text{PbSO}_4$	0.36	
	$\text{Co} \longrightarrow 2e^- + \text{Co}^{2+}$	0.28	
	$\text{Ni} \longrightarrow 2e^- + \text{Ni}^{2+}$	0.25	
	$\text{Sn} \longrightarrow 2e^- + \text{Sn}^{2+}$	0.14	
	$\text{Pb} \longrightarrow 2e^- + \text{Pb}^{2+}$	0.13	
	$\text{H}_2(\text{g}) \longrightarrow 2e^- + 2\text{H}^+$	0.00	
	$\text{H}_2\text{S}(\text{g}) \longrightarrow 2e^- + \frac{1}{8}\text{S}_8 + 2\text{H}^+$	-0.14	
	$\text{Sn}^{2+} \longrightarrow 2e^- + \text{Sn}^{4+}$	-0.15	
	$\text{Cu}^+ \longrightarrow e^- + \text{Cu}^{2+}$	-0.15	
	$\text{SO}_2(\text{g}) + 2\text{H}_2\text{O} \longrightarrow 2e^- + \text{SO}_4^{2-} + 4\text{H}^+$	-0.17	
	$\text{Cu} \longrightarrow 2e^- + \text{Cu}^{2+}$	-0.34	
	$\text{Cu} \longrightarrow e^- + \text{Cu}^+$	-0.52	
	$2\text{I}^- \longrightarrow 2e^- + \text{I}_2$	-0.53	
	$\text{H}_2\text{O}_2 \longrightarrow 2e^- + \text{O}_2(\text{g}) + 2\text{H}^+$	-0.68	

HALF-REACTION		E° (volts)
Reducing strength increases ↑	$\text{Fe}^{2+} \longrightarrow e^- + \text{Fe}^{3+}$	-0.77
	$\text{NO}_2(\text{g}) + \text{H}_2\text{O} \longrightarrow e^- + \text{NO}_3^- + 2 \text{H}^+$	-0.78
	$\text{Hg}(\text{l}) \longrightarrow 2 e^- + \text{Hg}^{2+}$	-0.78
	$\text{Hg}(\text{l}) \longrightarrow e^- + \frac{1}{2} \text{Hg}_2^{2+}$	-0.79
	$\text{Ag} \longrightarrow e^- + \text{Ag}^+$	-0.80
	$\text{H}_2\text{O} \longrightarrow 2 e^- + \frac{1}{2} \text{O}_2(\text{g}) + 2 \text{H}^+ (10^{-7} M)$	-0.815
	$\text{NO}(\text{g}) + 2 \text{H}_2\text{O} \longrightarrow 3 e^- + \text{NO}_3^- + 4 \text{H}^+$	-0.96
	$\text{Au} + 4 \text{Cl}^- \longrightarrow 3 e^- + \text{AuCl}_4^-$	-1.00
	$2 \text{Br}^- \longrightarrow 2 e^- + \text{Br}_2(\text{l})$	-1.06
	$\text{H}_2\text{O} \longrightarrow 2 e^- + \frac{1}{2} \text{O}_2(\text{g}) + 2 \text{H}^+$	-1.23
	$\text{Mn}^{2+} + 2 \text{H}_2\text{O} \longrightarrow 2 e^- + \text{MnO}_2 + 4 \text{H}^+$	-1.28
	$2 \text{Cr}^{3+} + 7 \text{H}_2\text{O} \longrightarrow 6 e^- + \text{Cr}_2\text{O}_7^{2-} + 14 \text{H}^+$	-1.33
	$2 \text{Cl}^- \longrightarrow 2 e^- + \text{Cl}_2(\text{g})$	-1.36
	$\text{Au} \longrightarrow 3 e^- + \text{Au}^{3+}$	-1.50
	$\text{Mn}^{2+} + 4 \text{H}_2\text{O} \longrightarrow 5 e^- + \text{MnO}_4^- + 8 \text{H}^+$	-1.52
Very weak reducing agents ↓	$\text{PbSO}_4 + 2 \text{H}_2\text{O} \longrightarrow 2 e^- + 4 \text{H}^+ + \text{SO}_4^{2-} + \text{PbO}_2$	-1.68
	$2 \text{H}_2\text{O} \longrightarrow 2 e^- + \text{H}_2\text{O}_2 + 2 \text{H}^+$	-1.77
	$2 \text{F}^- \longrightarrow 2 e^- + \text{F}_2(\text{g})$	-2.87
		Oxidizing strength increases ↓ Very strong oxidizing agents

Appendix 5

Solubility of Ionic Compounds in Water

Negative Ions (anions)	+ Positive Ions (cations)	form	Compounds which are :
All	Alkali ions (Li^+ , Na^+ , K^+ , Rb^+ , Cs^+)		Soluble*
All	Ammonium ion, NH_4^+		Soluble
Nitrate, NO_3^-	All		Soluble
Acetate, $\text{C}_2\text{H}_3\text{O}_2^-$	All		Soluble
Chloride, Cl^- Bromide, Br^- Iodide, I^-	Ag^+ , Pb^{2+} , Hg_2^{2+} , Cu^+ All others		Not soluble Soluble
Sulfate, SO_4^{2-}	Ba^{2+} , Sr^{2+} , Pb^{2+} , Ca^{2+} All others		Not soluble Soluble
Sulfide, S^{2-}	Alkali ions, NH_4^+ , Be^{2+} , Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+} All others		Soluble Soluble Not soluble
Hydroxide, OH^-	Alkali ions, NH_4^+ , Sr^{2+} , Ba^{2+} All others		Soluble Soluble Not soluble
Phosphate, PO_4^{3-} Carbonate, CO_3^{2-} Sulfite, SO_3^{2-}	Alkali ions, NH_4^+ All others		Soluble Not soluble

* Soluble means more than 0.1 mole will dissolve per liter.

Appendix 6

Some Mathematics Useful in Chemistry

EXPONENTIAL NUMBERS

Expressing Numbers in Exponential Notation

In chemistry we often have to deal with either very large numbers, such as

602,300,000,000,000,000,000,

the number of molecules in a mole, or very small numbers, such as

0.000 000 000 000 000 000 000 000 911 g,

the mass of an electron. Such numbers are much more conveniently expressed as some number between 1 and 10 times an exponential term. The exponential contains an exponent which is on a base. The base is usually 10. For example:

$1 = 1 \times 10^0$	$1 = \frac{1}{10^0} = 1$
$30 = 3 \times 10^1$	$0.2 = \frac{2}{10^1} = 2 \times 10^{-1}$
$500 = 5 \times 10^2$	$0.04 = \frac{4}{10^2} = 4 \times 10^{-2}$
$8000 = 8 \times 10^3$	$0.007 = \frac{7}{10^3} = 7 \times 10^{-3}$

The rules for manipulating exponentials are few and simple.

- 1) Multiplying by an exponential with a positive exponent shifts a decimal point to the right: $1.23 \times 10^2 = 123$.
- 2) Multiplying by an exponential with a negative exponent shifts a decimal point to the left: $4.56 \times 10^{-3} = 0.00456$.

- 3) If an exponential is shifted from numerator to denominator, or vice versa, the sign of the exponent changes.

$$10^2 = \frac{10^2}{1} = \frac{1}{10^{-2}}$$

$$\frac{1}{10^3} = \frac{10^{-3}}{1} = 10^{-3}$$

- 4) To multiply exponentials, simply add the exponents.

$$10^5 \times 10^2 = 10^7 \quad 10^3 \times 10^{-4} = 10^{-1}$$

- 5) To divide exponentials, simply subtract the exponents in the denominator from those in the numerator.

$$\frac{10^6}{10^4} = 10^2 \quad \frac{10^{-3}}{10^{-4}} = 10^{-3-(-4)} = 10^1$$

These rules explain why $10^0 = 1$. A number divided by itself equals one, $\frac{10^2}{10^2} = 1$. But also

$$\frac{10^2}{10^2} = 10^{2-2} = 10^0$$

So $10^0 = 1$.

In exponential notation,

$$602,300,000,000,000,000,000 = 6.023 \times 10^{23}$$

and

$$0.000\ 000\ 000\ 000\ 000\ 000\ 000\ 000\ 911 = 9.11 \times 10^{-28}$$

Writing a number in this form is a "shorthand" method of expression. The notation 6.023×10^{23}

tells us to multiply 6.023 by 10 twenty-three times. The notation 9.11×10^{-28} tells us to divide 9.11 by 10 twenty-eight times. Recall that to multiply by 10 moves the decimal point one place to the right and to divide by 10 moves the decimal point one place to the left.

This method of notation is extremely useful in indicating the number of significant figures in a measurement and eliminates the confusion that sometimes occurs as to whether or not zeros in a number are significant.

EXAMPLES

The value given above for the number of molecules in a mole is known to be reliable to four significant figures. It is therefore written in exponential form as 6.023×10^{23} molecules. The number 6.023 expresses the proper number of significant figures. The term 10^{23} indicates the magnitude of the term.

The numbers below are correctly written using exponential notation.

$$186,000 \pm 1,000 = 1.86 \times 10^5$$

(three significant figures)

$$0.00430 \pm 0.00001 = 4.30 \times 10^{-3}$$

(three significant figures)

$$10,002 \pm 1 = 1.0002 \times 10^4$$

(five significant figures)

EXERCISES

- 1) Make the indicated changes.

Examples:

$$224 \times 10^5 = 2.24 \times 10^7$$

Answer:

$$224 \times 10^5 = 2.24 \times 10^7.$$

$$3224 \times 10^{-5} = 3.224 \times 10^2$$

Answer:

$$3224 \times 10^{-5} = 3.224 \times 10^{-2}.$$

- $448 \times 10^4 = 4.48 \times 10^6$
- $0.035 \times 10^8 = 3.5 \times 10^9$
- $2.35 \times 10^4 = \underline{\hspace{1cm}} \times 10^5$
- $8.1 \times 10^6 = \underline{\hspace{1cm}} \times 10^4$

- $324.5 \times 10^9 = \underline{\hspace{1cm}} \times 10^{11}$
- $224 \times 10^{-5} = 2.24 \times 10^2$
- $0.045 \times 10^{-9} = 4.5 \times 10^9$
- $76.16 \times 10^{-4} = 7616 \times 10^{-2}$ (put the decimal point in the proper place)
- $3.1 \times 10^{-6} = \underline{\hspace{1cm}} \times 10^{-3}$
- $5.423 \times 10^{-9} = \underline{\hspace{1cm}} \times 10^{-11}$

- 2) Change each of the following to its non-exponential form. Assume there is an uncertainty of one in the last digit shown.

Examples:

$$2.24 \times 10^4 = 22,400 \pm 100;$$

$$4.30 \times 10^{-3} = 0.00430 \pm 0.00001.$$

- 3×10^2
- 8.5×10^4
- 1.86251×10^5
- 1.61×10^7
- 5×10^{-3}
- 9.4×10^{-6}
- 1.83×10^{-4}
- 1.55×10^{-2}
- 8.010×10^1
- 8.0×10^{-3}

- 3) Express the following in exponential notation. Each answer should consist of the product of a number between 1 and 10 which reflects the number of significant figures and a power of ten.

Examples:

$$22,400 \pm 100 = 2.24 \times 10^4;$$

$$0.0056 \pm 0.0001 = 5.6 \times 10^{-3}.$$

- 300 ± 100
- $85,000 \pm 1000$
- $186,251 \pm 1$
- $16,100,000 \pm 100,000$
- 0.005 ± 0.001
- 0.0000094 ± 0.0000001
- 0.000183 ± 0.000001
- 0.0155 ± 0.0001
- 0.0080 ± 0.0001
- 80.10 ± 0.01

Addition and Subtraction of Numbers in Exponential Notation

First express them to the same power of 10, then add or subtract the nonexponential term. Keep the exponential term.

EXAMPLES

$$\begin{aligned}(2.3 \times 10^4) + (1.4 \times 10^5) &= \\ (2.3 \times 10^4) + (14 \times 10^4) &= 16 \times 10^4 \\ (9.6 \times 10^5) - (3.2 \times 10^4) &= \\ (9.6 \times 10^5) - (0.32 \times 10^5) &= 9.3 \times 10^5\end{aligned}$$

EXERCISES

- 1) $(2.3 \times 10^4) + (4.6 \times 10^4) =$
- 2) $(2.3 \times 10^4) + (5.3 \times 10^5) =$
- 3) $(8.6 \times 10^{-3}) + (5.0 \times 10^{-4}) =$
 9.1×10^{-3} (answer)
- 4) $(2.18 \times 10^{-2}) - (1.09 \times 10^{-2}) =$
- 5) $(6.23 \times 10^{-9}) - (8.5 \times 10^{-10}) =$
- 6) $(8.6 \times 10^7) - (3.0 \times 10^6) =$
 8.3×10^7 (answer)

Multiplication of Numbers in Exponential Notation

First multiply the nonexponential terms; then add the exponents.

EXAMPLES

$$\begin{aligned}10^1 \times 10^2 \times 10^4 &= 10^{1+2+4} = 10^7 \\ 10^{-2} \times 10^3 \times 10^5 &= 10^{-2+3+5} = 10^6 \\ (1.0 \times 10^2)(2.2 \times 10^3)(1.5 \times 10^4) &= \\ 1.0 \times 2.2 \times 1.5 \times 10^{2+3+4} &= 3.3 \times 10^9\end{aligned}$$

EXERCISES

(Express your answer in correct exponential notation)

- 1) $10^2 \times 10^3 =$
- 2) $(2 \times 10^7)(2 \times 10^4) =$
- 3) $(1.2 \times 10^{12})(8.0 \times 10^8) =$
- 4) $(2.5 \times 10^8)(4.0 \times 10^{-3}) =$
- 5) $(1.3 \times 10^{-6})(3.0 \times 10^{-4}) =$

Division of Numbers in Exponential Notation

Divide the nonexponential terms; then subtract the exponents in the denominator from those in the numerator.

EXAMPLES

$$\begin{aligned}10^4 \div 10^1 &= 10^{4-1} = 10^3 \\ 8 \times 10^3 \div 2 \times 10^3 &= 8 \div 2 \times 10^{3-3} \\ &= 4 \times 10^0 = 4 \\ \frac{2 \times 10^3 \times 1 \times 10^{-2} \times 3 \times 10^6}{6 \times 10^{-6}} &= \\ \frac{2 \times 1 \times 3}{6} \times \frac{10^{3-2+6}}{10^{-6}} &= \frac{10^7}{10^{-6}} \\ &= 10^{7-(-6)} \\ &= 10^{13}\end{aligned}$$

EXERCISES

- 1) $10^6 \div 10^2 = 10^?$
- 2) $(2.7 \times 10^8) \div (3 \times 10^5) =$
- 3) $(4.5 \times 10^{-9}) \div (5 \times 10^4) =$
- 4) $(1.77 \times 10^8) \div (3 \times 10^{-3}) =$
- 5) $(1.45 \times 10^{-8}) \div (5.0 \times 10^{-12}) =$

Square Root of Numbers in Exponential Notation

Multiply the exponent by $\frac{1}{2}$ and obtain the square root of the other factor. If the exponent is not divisible evenly by two, change the nonexponential factor by moving the decimal point to the right or to the left, whichever is more convenient, and make the required change in the exponent.

EXAMPLES

$$\begin{aligned}\sqrt{y^4} &= (y^4)^{1/2} = y^2 \\ (4 \times 10^2)^{1/2} &= 4^{1/2} \times (10^2)^{1/2} = 2 \times 10 = 20 \\ (9 \times 10^4)^{1/2} &= 9^{1/2} \times (10^4)^{1/2} = 3 \times 10^2 = 300 \\ (2.5 \times 10^5)^{1/2} &= (25 \times 10^4)^{1/2} = \\ &= 25^{1/2} \times (10^4)^{1/2} = 5 \times 10^2 \\ (10 \times 10^9)^{1/2} &= (1.0 \times 10^{10})^{1/2} = \\ &= 1.0^{1/2} \times (10^{10})^{1/2} = 1 \times 10^5\end{aligned}$$

EXERCISES

(Express your answers in correct exponential notation)

- 1) $\sqrt{10^8} =$
- 2) $(2.5 \times 10^7)^{1/2} =$
- 3) $(62.5 \times 10^{-5})^{1/2} =$
- 4) $\sqrt{16} \times 10^{12} =$
- 5) $(16.9 \times 10^{11})^{1/2} =$

ADDING, SUBTRACTING, MULTIPLYING, AND DIVIDING EQUATIONS

Many of the fundamentals you have learned in algebra are extremely useful in chemistry. Some of the more commonly used manipulations are briefly reviewed here. Study each example as a means of refreshing your previous experience.

Consider two pairs of equations. The first pair you will recognize from your experience in algebra, while the second pair makes use of symbols common to chemistry. They are otherwise identical.

- a. $2a + b = 2c$
 $2c + b = 2e$
- b. $2\text{C} + \text{O}_2 = 2\text{CO}$
 $2\text{CO} + \text{O}_2 = 2\text{CO}_2$

When we add the equations we obtain

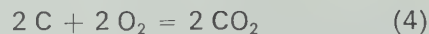
$$2a + 2b + 2c = 2c + 2e \quad (1)$$

$$2\text{C} + 2\text{O}_2 + 2\text{CO} = 2\text{CO} + 2\text{CO}_2 \quad (2)$$

The same quantity may be added or subtracted from each side of an equation without changing the equality. Subtracting $2c$ from each side of equation (1), we obtain

$$2a + 2b = 2e \quad (3)$$

Subtracting 2CO from each side of equation (2), we obtain



Each side of an equation may be divided by the same quantity without changing the equality. Dividing equation (3) by 2 we obtain

$$a + b = e$$

This is the simplified sum of the two equations in example a. Dividing equation (4) by 2 gives



which is the simplified sum of the two equations in example b.

Let us consider two more pairs of equations:

- c. $a + b = c + 26d$
 $e + b = c + 24d$
- d. $\text{C(diamond)} + \text{O}_2(\text{g}) =$
 $\text{CO}_2(\text{g}) + 94.50 \text{ kcal}$
 $\text{C(graphite)} + \text{O}_2(\text{g}) =$
 $\text{CO}_2(\text{g}) + 94.05 \text{ kcal}$

Subtracting one equation from the other we obtain:

$$a - e = 2d \quad (5)$$

and

$$\text{C(diamond)} - \text{C(graphite)} = 0.45 \text{ kcal} \quad (6)$$

Adding e to each side of equation (5) and C(graphite) to each side of equation (6), we obtain:

$$a = e + 2d$$

and

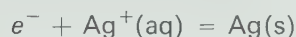
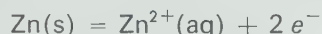
$$\text{C(diamond)} = \text{C(graphite)} + 0.45 \text{ kcal}$$

EXERCISES

- 1) Given the following two steps for the production of SO_3 , add these equations to get the equation for the production of SO_3 from S_8 and O_2 .

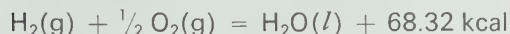
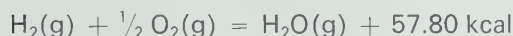


- 2) Add the following two equations so that e^- cancels out.



- 3) Energy is required to evaporate water. The following equations indicate the energy released when hydrogen and oxygen are burned to form water. Find the energy required to evaporate $\text{H}_2\text{O}(\text{l})$.

[Hint: _____ kcal + $\text{H}_2\text{O}(\text{l}) = \text{H}_2\text{O}(\text{g})$.]



GRAPHING

Many properties are found to be interdependent. As the temperature of a gas increases, the volume increases if other conditions are not changing. Such a relation in which two values change in the same direction (one remains larger than the other by the same factor) is a **direct relation**. A direct relation may be expressed mathematically as $x = ky$ or $x/y = k$, where k is a constant. Consider the following example of the direct relation between the volume of a gas, V , and its absolute temperature, T , where $V = kT$. Note the straight line which results when the values are plotted. See Figure A6-1.

Temperature (°K)	Volume (liters)
120	1
240	2
360	3

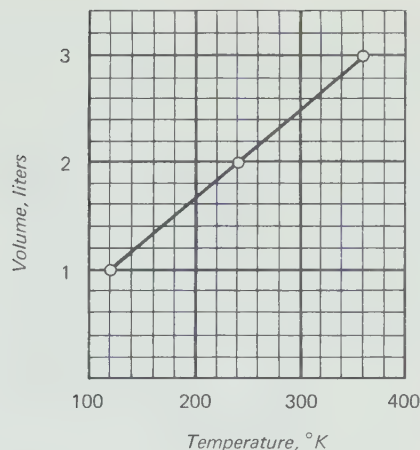


Figure A6-1

Graph for $V = kT$.

MORE EXAMPLES

- a. If $k = 1$, we find that $x = y$. As x increases in value y also increases. Plotting these values on a graph with values of x along the horizontal axis (abscissa) and y along the vertical axis (ordinate) we obtain Figure A6-2.

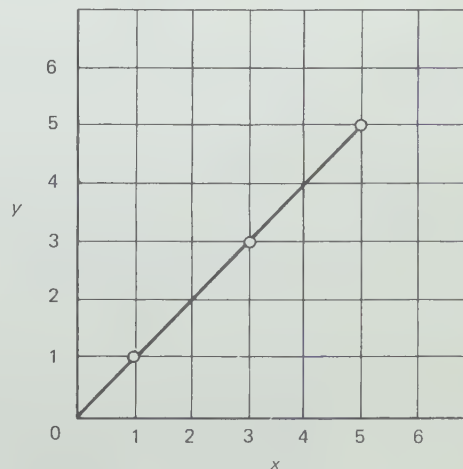


Figure A6-2

Graph for $x = y$.

x	y
1	1
3	3
5	5

- b. If $k = 2$, we find that $x = 2y$. Plotting these values, we obtain Figure A6-3.

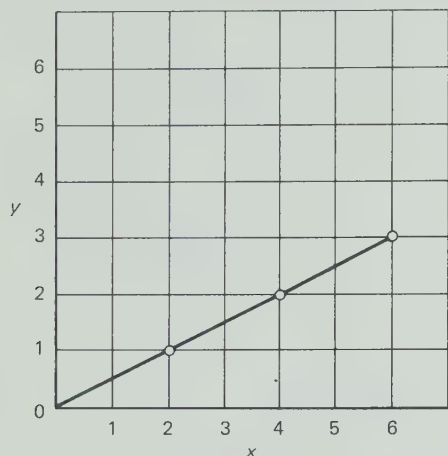


Figure A6-3

Graph for $x = 2y$.

x	y
2	1
4	2
6	3

Note that in either case the plot is a straight line passing through the origin where $x = y = 0$.

Consider now another kind of interdependence. When one value becomes larger by a given factor and the other becomes smaller by the same factor, we have an **inverse relation**, which is expressed mathematically as $xy = k$ or $y = k/x$.

If $k = 6$, we find these values for x and y .

x	y
1	6
2	3
3	2
6	1

Plotting these values we obtain Figure A6-4. The relation between gas pressure and volume discussed in Chapter 1 of the Textbook (page 10) is an example of an inverse relation in which $P = k/V$ or $PV = k$. Note the type of curve which results when the data for the pressure

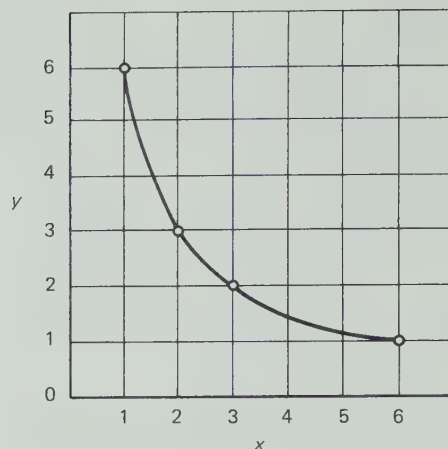


Figure A6-4

Graph for $xy = 6$.

and volume of 32.0 grams of oxygen gas are plotted in Figure A6-5.

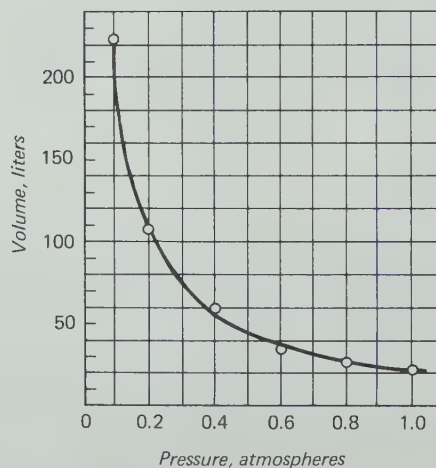


Figure A6-5

Graph for $PV = k$.

Pressure (atmospheres)	Volume (liters)
0.100	224
0.200	109
0.400	57.5
0.600	38.0
0.800	27.7
1.00	22.4

Note that in an inverse relation, for any value of k (except $k = 0$), when x becomes very large, y becomes very small and approaches but never reaches zero. When y becomes very large, x becomes very small but never reaches zero.

EXERCISES

- 1) Plot four values of x and y in which the relation is direct and $k = 3$.
- 2) Plot six values of x and y in which the relation is inverse and $k = 3$. Start with $x = 1$ and $y = 3$.
- 3) Determine the value for k from the following data :

x	y
0.2	0.4
0.6	1.2
1.8	3.6

Plot these values.

- 4) The volume of a partially inflated rubber balloon varies inversely with the external pressure.
 - a. Write an equation to describe this relation. Use P for pressure and V for volume.
 - b. It is found experimentally that when the pressure is 1 atmosphere the balloon has a volume of 22.4 liters. When the pressure is increased to 2 atmospheres the volume decreases to 11.2 liters. What is the value of k in the equation?
 - c. What would the volume be under a pressure of 12 atmospheres? Of $\frac{1}{2}$ atmosphere?

Appendix 7

Uncertainty in Measurement

When you measure a board, you might say, "It's nearer $12\frac{3}{16}$ inches than $12\frac{1}{4}$; I'll call it $12\frac{3}{16}$ inches." The board is a little more than $12\frac{3}{16}$ inches long. Another time, you might measure the board and decide it is closer to $12\frac{1}{4}$ inches long. With each measurement, there is a small amount of uncertainty. Using an ordinary ruler, you can determine length to within about $\frac{1}{32}$ inch. A micrometer can be used to determine lengths to the nearest $1/1000$ of an inch. In all cases, there is some uncertainty associated with each measurement. Good instruments and skillful users can reduce but not eliminate uncertainty.

The word *precision* is used in discussing the variations found when repeated measurements

are made using the same procedure. If repeated weighings gave the mass of an object as 1.23, 1.25, 1.24, 1.24, and 1.25 grams, the precision would be quite satisfactory for a centigram balance. The mass should be reported as 1.24 ± 0.01 grams. If, on the other hand, the weighings were 1.23, 1.27, 1.20, 1.31, and 1.33 grams, the precision is poor. The balance or the balance user needs some attention. Such variation is caused by limitations of both the measuring device and the experimenter's ability to use the device.

The following table shows typical uncertainty values associated with the apparatus generally used in this course. Gross errors due to blunders or lack of knowledge of proper procedures for making observations are not included.

UNCERTAINTIES ASSOCIATED WITH MEASUREMENTS
MADE USING VARIOUS INSTRUMENTS

Instrument	Typical Uncertainty
Centigram balance	± 0.01 gram
Platform balance	± 0.5 gram
50 ml graduated cylinder	± 0.5 ml
10 ml graduated cylinder	± 0.1 ml
-20 to 150°C thermometer	± 0.2 C°
50 ml buret	± 0.02 ml
50 ml gas measuring tube	± 0.02 ml

An important step when recording data is to record the uncertainty also. For example, if you weigh a piece of copper wire on a centigram balance, you might record

Mass of copper wire: $2.89 \text{ g} \pm 0.01 \text{ g}$

The " $\pm 0.01 \text{ g}$ " indicates the weighing was done on a balance that would show the difference of

0.01 gram. Most repeated weighings on this balance would be in the range 2.88 to 2.90. The average would be 2.89.

Another way to express the uncertainty is to indicate its magnitude as a percent of the measured quantity. For the example given above, the percent uncertainty is

$$\frac{0.01 \text{ g}}{2.89 \text{ g}} \times 100 = 0.35\%$$

So the measurement could be recorded as

Mass of copper wire: $2.89 \text{ g} \pm 0.35\%$

This method has several advantages when we consider how errors affect calculated results which involve multiplication or division. It is also a clearer way to compare uncertainties in quantities of different magnitude. For example, a much larger piece of copper might weigh $28.92 \text{ g} \pm 0.01 \text{ g}$. The percent uncertainty is

$$\frac{0.01 \text{ g}}{28.92 \text{ g}} \times 100 = 0.035\%$$

This mass of copper could be recorded as

$28.92 \text{ g} \pm 0.035\%$

The absolute uncertainty in the two cases is the same: 0.01 g. However, there is a greater degree of uncertainty in the first example. If this second piece of copper were weighed on a less sensitive device such as a platform balance, we might have found the mass to be $28.9 \text{ g} \pm 0.5 \text{ g}$ or $28.9 \text{ g} \pm 1.7\%$, which is a much less precise value.

A third way of indicating the uncertainty is by the number of figures recorded. This is known as the method of "significant figures," and although it is often convenient it does not give as much information as the two preceding methods. In spite of its limitations, the method using significant figures is satisfactory for most purposes in this course.

In this method all digits that are certain and one additional uncertain digit are shown. All of these digits are "significant figures," or "sig figs" for short. For example, for the copper wire weighed on the centigram balance we recorded

$2.89 \pm 0.01 \text{ g}$; so this mass would be written with three significant figures, 2.89 g. Note that the amount by which the last digit is uncertain is not specified when using only the significant figure notation. The numbers $2.89 \pm 0.02 \text{ g}$, $2.89 \pm 0.05 \text{ g}$ or $2.89 \pm 0.09 \text{ g}$ would also be written as 2.89 grams.

The large piece of copper wire weighed on a platform balance had a mass of $28.9 \text{ g} \pm 0.5 \text{ g}$. Using the sig fig notation, this would be written 28.9 g, showing three significant figures. Here again we are not sure how large the uncertainty is.

THE USE OF SIGNIFICANT FIGURES IN RECORDING MEASUREMENTS

- 1) The integers recorded are those that are certain and one more in which there is some uncertainty. Note the examples above.
- 2) The number of significant figures has nothing to do with location of the decimal point. Thus, zeros that merely indicate the magnitude of the number are not significant figures.

For example, each of the numbers 36.09, 3.609, 0.3609, 0.003609, and 3609 has four significant figures. The number 36.090 has five significant figures, but what about the number 36090? In this case the last 0 may be a significant figure or it may merely mark the decimal place. To make it entirely clear that the last 0 is significant, the number should be written as 3.6090×10^4 . To indicate that the last zero is not significant, write 3.609×10^4 .

Note again that using the number of significant figures to indicate uncertainty assumes that the uncertainty is always in the last digit — it does not tell how much uncertainty there is.

THE PROPAGATION OF ERRORS IN CALCULATED RESULTS

So far we have discussed only the problems connected with recording a single measurement with the proper notation of uncertainty. Now we will consider how the uncertainties of several individual measurements carry over and combine when making various kinds of calculations to give us a derived result and its uncertainty.

A. Addition and Subtraction

When quantities are added or subtracted the maximum uncertainty in the result is the sum of the uncertainty for each of the component measurements.

RESULT DERIVED BY SUBTRACTION, EXAMPLE A

In Experiment 7 you obtained the mass of silver nitrate, $\text{AgNO}_3(\text{s})$, by making two weighings such as these:

Mass of vial and $\text{AgNO}_3(\text{s})$	$15.34 \pm 0.01 \text{ g}$
Mass of vial	$12.83 \pm 0.01 \text{ g}$
Mass of $\text{AgNO}_3(\text{s})$	$2.51 \pm 0.02 \text{ g}$

Note that the uncertainty in the derived result is $\pm 0.02 \text{ g}$, or the sum of the individual uncertainties. Let us check this by using the values which will give the greatest and the least differences:

Greatest		Least	
$(15.34 + 0.01)$	15.35 g	$(15.34 - 0.01)$	15.33 g
$(12.83 - 0.01)$	12.82 g	$(12.83 + 0.01)$	12.84 g
Difference	2.53 g		2.49 g

These numbers are in accord with the notation $2.51 \pm 0.02 \text{ grams}$.

RESULT DERIVED BY ADDITION, EXAMPLE B

In Experiment 8 you weighed out a sample of sodium chloride, $\text{NaCl}(\text{s})$, with an uncertainty of $\pm 0.02 \text{ g}$. Later you added this mass to the mass of silver nitrate, $\text{AgNO}_3(\text{s})$, also known with an uncertainty of $\pm 0.02 \text{ g}$:

Mass of $\text{NaCl}(\text{s})$	$0.87 \pm 0.02 \text{ g}$
Mass of $\text{AgNO}_3(\text{s})$	$2.51 \pm 0.02 \text{ g}$
Sum	$3.38 \pm 0.04 \text{ g}$

Note that the uncertainty in the derived result is $\pm 0.04 \text{ g}$. Check this by finding the greatest and least values for the sum, as was done in Example A.

EXAMPLES USING THE SIGNIFICANT FIGURE METHOD

In Example A, only three significant figures can be used to express the difference, 2.51 g , since the uncertainties involved occur in the hundredths place. This means that the first two digits are known with certainty, but there is some question about the last digit. Note that in subtraction (or addition) the answer will not always have the same number of significant figures as in the data (four in Example A). You must observe in which position, tenths, hundredths, etc., the digit with "doubt" occurs.

Consider a more extreme example. Add:

$27 \text{ cm} \pm 1 \text{ cm}$	uncertainty in the units place
$2.35 \text{ cm} \pm 0.01 \text{ cm}$	uncertainty in the hundredths place

The former value limits the answer to $29 \pm 1 \text{ cm}$ or to two significant figures.

Note that the method of using significant figures is superficially simple but does not communicate the magnitude of the uncertainty. Nevertheless, it is adequate for most of the calculations we shall make.

EXERCISES

Calculate the maximum uncertainty in each of the following.

<i>Subtraction</i>		<i>Addition</i>	
1)	$40.2 \pm 0.2^\circ\text{C}$ $- 10.2 \pm 0.2^\circ\text{C}$	2)	$1500 \pm 10 \text{ cm}$ $1500 \pm 10 \text{ cm}$ $481 \pm 1 \text{ cm}$ $+ 481 \pm 1 \text{ cm}$
3)	$103.24 \pm 0.01 \text{ g}$ $- 98.13 \pm 0.01 \text{ g}$	4)	$5.18 \pm 0.02 \text{ g}$ $+ 1.76 \pm 0.02 \text{ g}$

B. Multiplication and Division

In multiplication and division the derived uncertainty is not simply the sum of the uncertainties in the factors. It is the sum of the *percent* uncertainties in the factors.

EXAMPLE

Calculate the ratio of moles of aluminum to moles of silver if the moles of aluminum are 1.49 ± 0.02 and the moles of silver are 0.51 ± 0.02 .

$$\frac{\text{moles of aluminum}}{\text{moles of silver}} = \frac{1.49 \pm 0.02}{0.51 \pm 0.02} = \frac{1.49 \pm 1.3\%}{0.51 \pm 3.9\%}$$

Answer: $2.92 \pm 5.2\%$ or 2.92 ± 0.15 .

When using significant figures we assume that the product or quotient will have the same number of significant figures as the least precise component. This is not an exact rule but is often convenient to use.

Using significant figures, we have

$1.49 =$ three significant figures

$0.51 =$ two significant figures

The answer should have two significant figures so 2.92 is rounded off to 2.9 . Our easy-to-use-rule gives less certainty than the data warrants.

EXERCISES

- 5) Find the area of a rectangle measured to be 10.0 ± 0.1 cm by 2.5 ± 0.1 cm, and calculate the uncertainty by each method (by using significant figures and by summing of percent uncertainty). Make a drawing of the rectangle, shading in the area of uncertainty. Calculate the area of the shaded part, and compare this to the calculated uncertainty.
- 6) Find the heat absorbed if $200 \text{ g} \pm 0.5 \text{ g}$ of water is heated $5.0 \pm 0.2^\circ\text{C}$. (One calorie of energy is required to change the temperature of one gram of water by 1 Celsius degree.)
- 7) Find the heat of combustion (calories per gram) for a substance if 9800 ± 200 calories are liberated when 1.23 ± 0.02 g of it burns. Express your answer to the correct number of significant figures.
- 8) Calculate the heat of solidification for the above substance if 210 ± 70 calories of heat are liberated as 10.3 ± 0.2 g of it solidifies. Express your answer to the correct number of significant figures.

ROUNDING OFF NUMBERS

Calculated results sometimes contain more digits than are justified from the uncertainty in the data. For example, find the area of a rectangle 15.2 ± 0.1 cm by 13.2 ± 0.1 cm. The product, neglecting uncertainty, is 200.64 cm^2 . Since the least precise factor contains 3 sig figs, we round the answer to 201 cm^2 .

If the number 56.24 is to be rounded off to three digits, you would use 56.2 since 0.24 is nearer 0.2 than 0.3 . Also:

234.57 rounded off to four digits is 234.6 ;

234.12 rounded off to four digits is 234.1 ;

234.546 rounded off to five digits is 234.55 ;

234.546 rounded off to four digits is 234.5 .

When only a 5 is dropped the last digit remaining is changed to the next higher even number if this digit is odd, and is left unchanged if it is even.

EXAMPLE

234.75 is rounded off to 234.8 ;

234.45 is rounded off to 234.4 .

This is done to minimize the accumulated error caused by rounding. For example, when a 9 is dropped, we effectively add 1 when the last remaining digit is increased. This increase is however cancelled when a 1 is dropped. Since a 9 is just as likely to occur as a 1, these two digits cause no accumulation of error. The same situation prevails when dropping an

8 or a 2,
a 7 or a 3,
a 6 or a 4.

In all three cases, the gain is cancelled by an equally probable loss. However, we are left with nothing to match the 5 against. So a match is created. The digit before a to-be-dropped 5 is just as likely to be odd as even. If we add in one case and drop the same amount in the other case, our errors will cancel. The rule is to finish with an even digit — the world likes even things. If the last remaining digit is even, we rejoice and

throw away the 5. If the last remaining digit is odd, we groan and add five to make it even.

ACCURACY OF A RESULT

The words precision and accuracy do not refer to the same aspects of measurement. Precision refers to internal agreement. Accuracy refers to agreement with an external standard.

Ten weighings of an object on one balance might all be within the range 23.65 ± 0.02 grams. The precision is quite acceptable for a centi-

gram balance. But suppose another, more sensitive balance gave for the same object a set of ten weighings, all of which were within the range 23.152 ± 0.002 grams. If a third balance gave a set of ten weighings all within the range 23.154 ± 0.002 grams, then the first balance would be regarded with suspicion. Perhaps checking it would show one of the notches being used was cut incorrectly so that weighings were consistently 0.50 gram low. This balance was giving high precision, but low accuracy.

Appendix 8

Working with Glass

TO CUT TUBING

Hold the glass tubing firmly between the thumb and an edge of a triangular file as shown in Figure A8-1. Rotate the glass about one-fourth of a turn to make a short, sharp scratch on the glass tubing.

Place your thumbs together, opposite the scratch; pull and bend quickly to snap the glass tubing at the scratch.

TO FIRE POLISH TUBING

To melt (fire-polish) the freshly cut edges, hold one end of the glass tubing in the hottest part of a burner flame and rotate the tube clockwise and counterclockwise until the edges are rounded. See Figure A8-2. Do not fire-polish so long that the end of the tube begins to close. **Caution:** Hot glass looks like cool glass. Put the hot glass on a wire gauze to cool, then fire-polish the other end of the tubing. A good treatment for burned fingers is to soak them immediately in cold water.

TO REDUCE TUBING DIAMETER

Heat the glass tubing in a burner flame, in the position shown in Figure A8-3, rotating the glass in the hottest part of the flame. Allow the tubing to become slightly shorter as the glass melts and the walls thicken to about twice their original thickness. After the glass becomes quite soft and

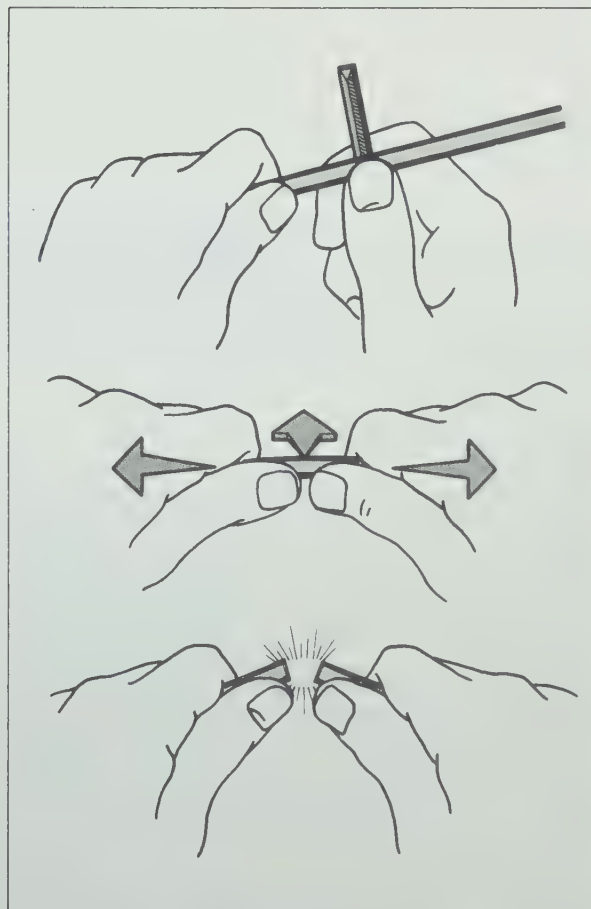


Figure A8-1
Cutting glass tubing.

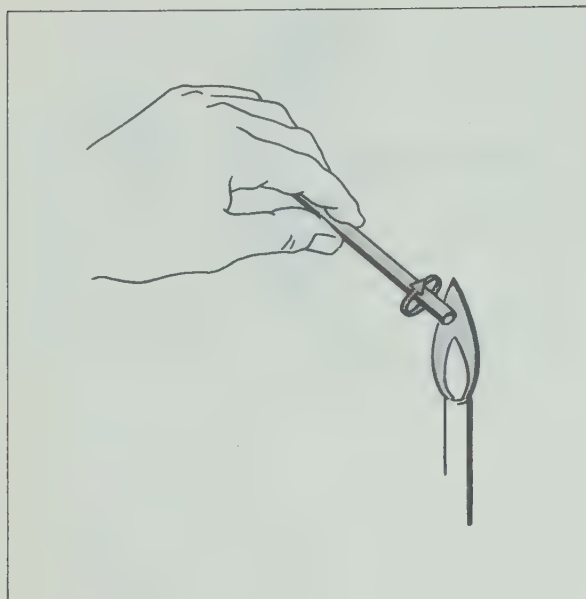


Figure A8-2
Fire-polishing glass tubing.

thickened, remove it from the flame and pull the tubing vertically until the desired diameter (jet tip) is obtained. Place the hot glass on a wire gauze to cool. Cut the glass in the middle of the drawn-out portion and fire-polish the sharp ends.

TO BEND TUBING

Heat the glass tubing by rotating it back and forth in a flat burner flame, using a wing top as shown in Figure A8-4. Continue heating the tubing until the glass becomes quite soft and is just beginning to sag as it is rotated. Remove the tubing from the burner flame and bend it by raising the ends to obtain the desired angle. Since hot glass tends to sag, bending it in this manner will produce a more uniformly smooth bend. Figure A8-4 also contrasts the difference between a "good" bend (upper bend) and two "bad" bends. The middle bend resulted from holding the glass too low in the flame so that the middle of the tubing was in the cool portion of the flame. The bottom bend was produced by concentrating the flame on too narrow a portion

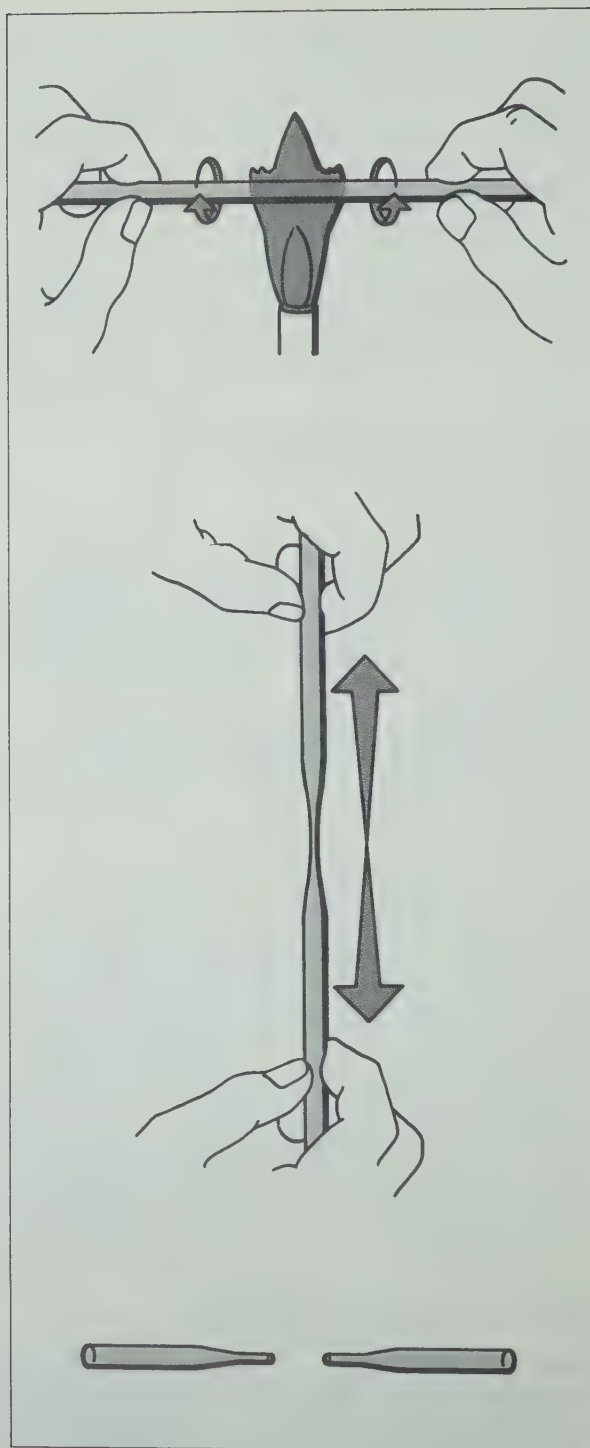
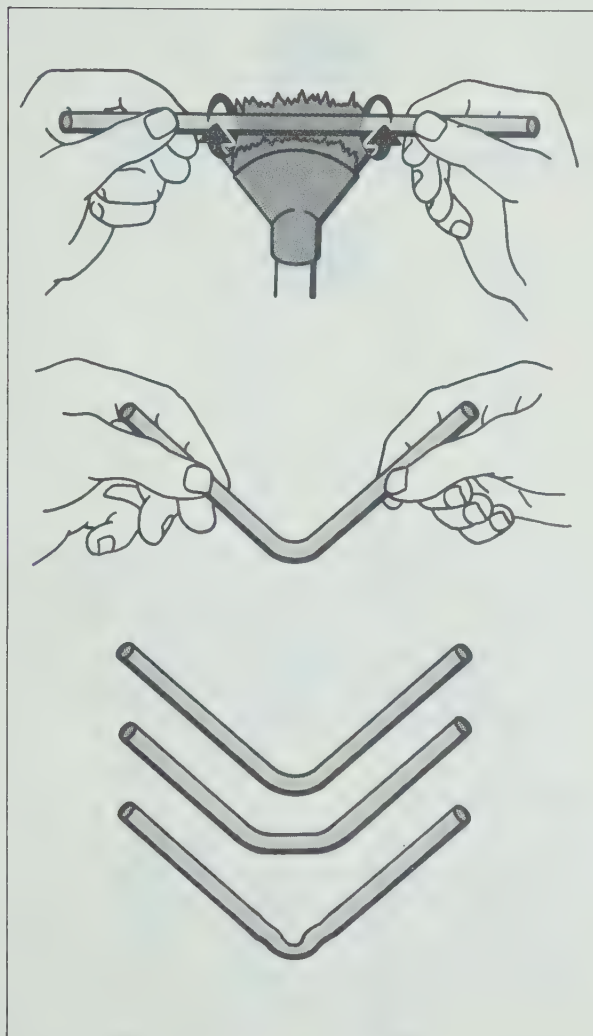


Figure A8-3
Drawing glass tubing to form a jet tip.

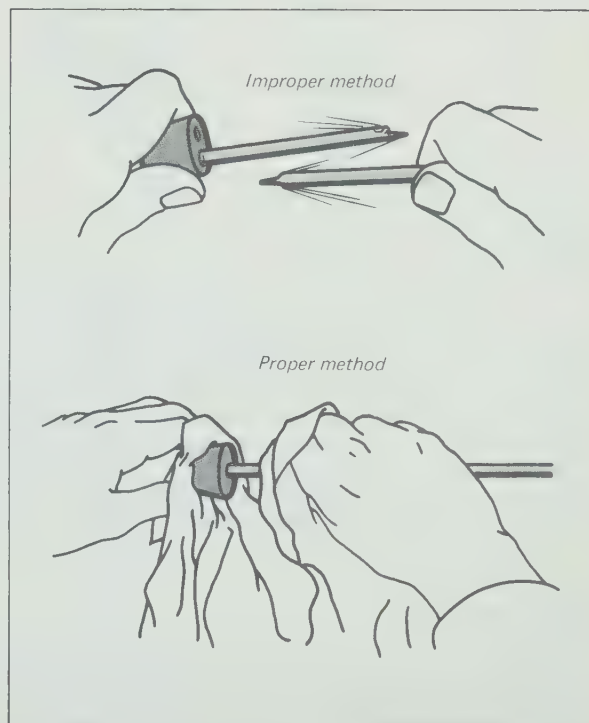
**Figure A8-4**

Making a smooth 90° bend in glass tubing.

of the glass tubing, which frequently results when a wing top is not used to spread the flame.

INSERTING GLASS TUBING OR A THERMOMETER INTO A RUBBER STOPPER

Before attempting to insert glass tubing into a rubber stopper be sure that the hole in the stopper is large enough that it will stretch easily to

**Figure A8-5**

Inserting glass tubing into a stopper.

accommodate the tubing. A drop of glycerol or some water placed in the hole of the stopper or on the tip of the glass will serve as a lubricant.

Protect your hands by wrapping the glass with a piece of cloth or towel. Hold the glass near the stopper and push it with a gentle twisting motion. See Figure A8-5. Do not force excessively as you insert glass tubing or a thermometer. **Be very careful.** If you experience any difficulty, consult your teacher.



Appendix 9

Care of Burets

Cleaning

Place a few milliliters of a detergent solution into a buret; then use a buret brush to clean the inside surface. Rinse well, first with tap water and then with distilled water. After draining the buret, note if there are any droplets still adhering to the inside of the tube. If there are, the glass is not thoroughly cleaned and should be rewashed. When glass is clean, water wets it evenly and drains off uniformly.

Preparing for Use

After cleaning a buret, add 2–3 ml of the solution that is to be used in that buret. Let some solution flow through the tip. Turn the buret to a horizontal position and slowly rotate the tube to make sure the solution wets the inside completely. Drain the solution out the tip. For a more complete rinsing, repeat the above.

Fill the buret with the solution to be used. Permit air bubbles in the tip to escape by turning the tip upward as shown in Figure A9-1. Let solution flow from the tip until the bottom of the meniscus is at zero or below.

If a drop hangs on the tip before you start a titration, discard the drop by touching it to a beaker. However, a drop formed **during** a titration must be caught by touching it to the side of the container being used and rinsing it into the container with distilled water.

Reading the Volume

When reading the volume on the buret, be sure to have your eye level with the bottom of the meniscus and read the volume carefully at the bottom of the curve (see Figure A9-2). In each titration use as large a volume as is practical. This helps reduce the percent error.

After Use

Drain and rinse the buret several times with tap water, then, as a final rinse, use distilled water. Glass reacts with basic solutions; so take special care in rinsing a buret that has had such solutions in it. A rinse with dilute acid after one water rinse will help assure that the base is removed. Follow this with the water rinses described above.

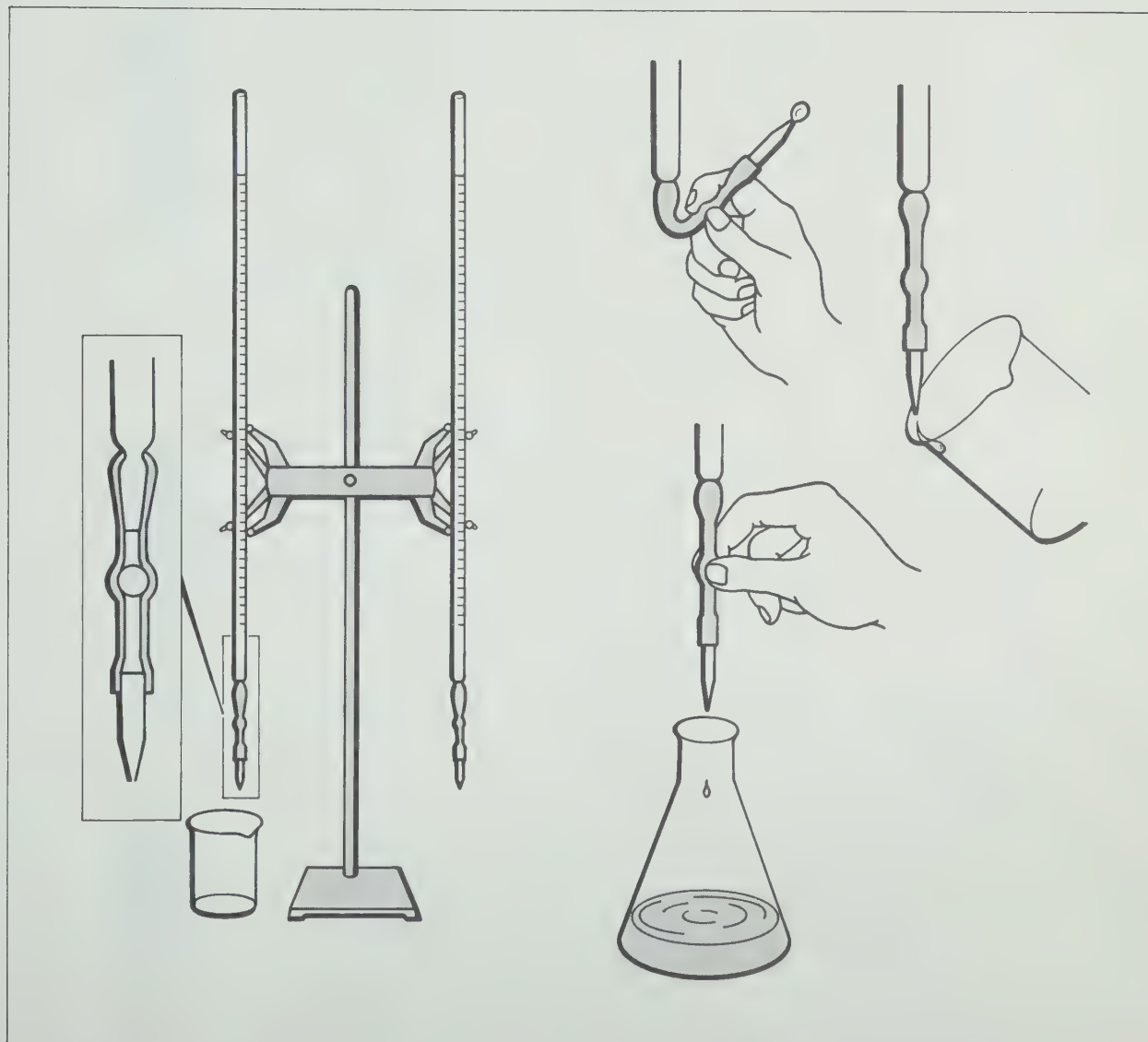


Figure A9-1 Buret assembly and use.

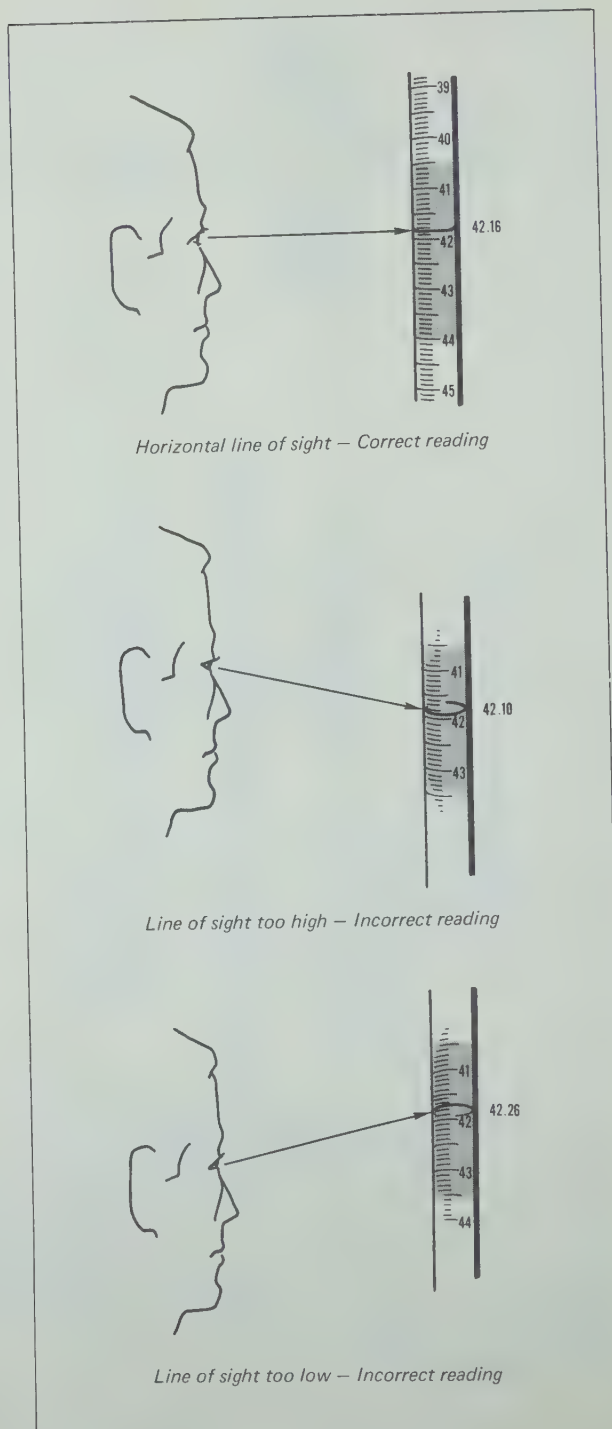


Figure A9-2
Reduce error by reading bottom
of meniscus with eye at proper level.

INTERNATIONAL MOLAR MASSES

NAME	SYMBOL	ATOMIC NUMBER	MOLAR MASS	NAME	SYMBOL	ATOMIC NUMBER	MOLAR MASS
Actinium	Ac	89	(227)	Mercury	Hg	80	200.6
Aluminum	Al	13	27.0	Molybdenum	Mo	42	95.9
Americium	Am	95	(243)	Neodymium	Nd	60	144.2
Antimony	Sb	51	121.8	Neon	Ne	10	20.2
Argon	Ar	18	39.9	Neptunium	Np	93	(237)
Arsenic	As	33	74.9	Nickel	Ni	28	58.7
Astatine	At	85	(210)	Niobium	Nb	41	92.9
Barium	Ba	56	137.3	Nitrogen	N	7	14.01
Berkelium	Bk	97	(247)	Nobelium	No	102	(253)
Beryllium	Be	4	9.01	Osmium	Os	76	190.2
Bismuth	Bi	83	209.0	Oxygen	O	8	16.00
Boron	B	5	10.8	Palladium	Pd	46	106.4
Bromine	Br	35	79.9	Phosphorus	P	15	31.0
Cadmium	Cd	48	112.4	Platinum	Pt	78	195.1
Calcium	Ca	20	40.1	Plutonium	Pu	94	(242)
Californium	Cf	98	(251)	Polonium	Po	84	210
Carbon	C	6	12.01	Potassium	K	19	39.1
Cerium	Ce	58	140.1	Praseodymium	Pr	59	140.9
Cesium	Cs	55	132.9	Promethium	Pm	61	(145)
Chlorine	Cl	17	35.5	Protactinium	Pa	91	(231)
Chromium	Cr	24	52.0	Radium	Ra	88	(226)
Cobalt	Co	27	58.9	Radon	Rn	86	(222)
Copper	Cu	29	63.5	Rhenium	Re	75	186.2
Curium	Cm	96	(248)	Rhodium	Rh	45	102.9
Dysprosium	Dy	66	162.5	Rubidium	Rb	37	85.5
Einsteinium	Es	99	(254)	Ruthenium	Ru	44	101.1
Erbium	Er	68	167.3	Samarium	Sm	62	150.4
Europium	Eu	63	152.0	Scandium	Sc	21	45.0
Fermium	Fm	100	(253)	Selenium	Se	34	79.0
Fluorine	F	9	19.0	Silicon	Si	14	28.1
Francium	Fr	87	(223)	Silver	Ag	47	107.9
Gadolinium	Gd	64	157.2	Sodium	Na	11	23.0
Gallium	Ga	31	69.7	Strontium	Sr	38	87.6
Germanium	Ge	32	72.6	Sulfur	S	16	32.1
Gold	Au	79	197.0	Tantalum	Ta	73	180.9
Hafnium	Hf	72	178.5	Technetium	Tc	43	(99)
Helium	He	2	4.00	Tellurium	Te	52	127.6
Holmium	Ho	67	164.9	Terbium	Tb	65	158.9
Hydrogen	H	1	1.008	Thallium	Tl	81	204.4
Indium	In	49	114.8	Thorium	Th	90	232.0
Iodine	I	53	126.9	Thulium	Tm	69	168.9
Iridium	Ir	77	192.2	Tin	Sn	50	118.7
Iron	Fe	26	55.8	Titanium	Ti	22	47.9
Krypton	Kr	36	83.8	Tungsten	W	74	183.9
Lanthanum	La	57	138.9	Uranium	U	92	238.0
Lawrencium	Lw	103	(259)	Vanadium	V	23	50.9
Lead	Pb	82	207.2	Xenon	Xe	54	131.3
Lithium	Li	3	6.94	Ytterbium	Yb	70	173.0
Lutetium	Lu	71	175.0	Yttrium	Y	39	88.9
Magnesium	Mg	12	24.3	Zinc	Zn	30	65.4
Manganese	Mn	25	54.9	Zirconium	Zr	40	91.2
Mendelevium	Md	101	(256)	(unnamed)*	?	104	(260)

* Discovery reported in 1964.

Molar masses based on $^{12}\text{C} + 12.000$.

Numbers in parentheses give the mass number of the most stable isotope.

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